



Material behaviour

Evaluation of nanotribological behavior of amorphous polystyrene: The macromolecular weight effect



Achraf Ghorbal*, Ammar Ben Brahim

Applied Thermodynamics Research Unit (UR11ES80E), National Engineering School of Gabès, Gabès University, Rue Omar Ibn-Elkhattab, 6029 Gabès, Tunisia

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ABSTRACT

Tribological testing of polymers is of prime importance in many industrial applications. Silicon nitride AFM tips have been used to mimic the contact between amorphous polystyrene surfaces and a hard asperity, which is useful in understanding of how a multitude of asperities behave in a macroscopic contact. In this study, the adhesion force and the friction force of four PS molecular weights were measured and the average contact pressure was calculated by using the JKR contact theory. The nanotribological behavior of polystyrene showed a dependence on macromolecular weight with varying applied normal force and sliding velocity. The study indicates that the length of polymer chains noticeably influences the tribological behavior of amorphous polystyrenes. Mechanisms governing such behavior differences were ascribed to energy dissipating modes.

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1. Introduction

Recent progress in tribological tests has been due in large part to experimental instruments giving access to the molecular and the atomic scales, such as the Surface Forces Apparatus (SFA) [1,2], the quartz-crystal microbalance [3] and the Atomic Force Microscope (AFM) [4]. Indeed, nanofrictional phenomena investigations offer a new insight into the behavior of systems typically considered to be 'macroscopic'. For example, contact between solid surfaces is generally made through a multitude of asperities that constitute the microscopic roughness of any 'real' surface [5–7]. Moreover, the understanding of frictional mechanisms at the interface is of prime importance in industrial applications, such as chemical engineering processes. In fact, engineering thermoplastics such as polystyrene [8–11] are widely used in the field of chemical engineering, e.g. processes in fluidized beds [12,13], powder technology [14],

materials handling and transport processing [15–17], biopolymers and polymers processing [18]. However, the whole description of chemical, physical, rheological and interfacial origins of polymers damage is not yet entirely available [19–21]. Furthermore, because of the important surface to volume ratio of surface asperities, the interfacial zone becomes increasingly important. In fact, the interface zone is expected to be the field of energy dissipation processes caused by the contribution of surface energy [22], and the mobility of macromolecules in the vicinity of the interface [23,24].

Clearly then, information about the tribological behavior of a single asperity must be useful in understanding of how a multitude of asperities will behave [25,26]. In this case, AFM has some advantages over other small-scale techniques in that the interactions between the AFM tip and a substrate can mimic a single-nanoscale asperity [27].

The earliest studies using AFM to probe frictional forces between materials lead to the development of the friction force microscopy (FFM), also called lateral force microscopy (LFM). As the tip slides across the surface at a given load, frictional forces acting between the tip and sample surface

* Corresponding author. Tel.: +216 75 392 100; fax: +216 75 392 190.
E-mail address: achraf.ghorbal.issat@gmail.com (A. Ghorbal).

can be detected by measuring the torsional bending of the cantilever, during FFM experiments. The adhesion between the tip and the surface can also be probed through force-distance measurements. Here, the tip is brought in contact and pressed against the surface to a fixed load and then retracted from the surface. A significant advantage of AFM as a nanotribological tool is its ability to investigate most types of materials (e.g. metals, ceramics, polymers) under a range of environments from ambient gas surroundings to vacuum or liquids [28–30].

The frictional behavior of glassy polymeric materials is expected to be significantly influenced by experimental and structural parameters.

The Leung et al. study [31] report that SFM tip multiline scanning produced a persistently periodic patterned structure perpendicular to the scanning direction on a polystyrene surface. This result demonstrated that there was no molecular weight dependence of the PS morphology. However, Meyers et al. [32] who studied the molecular weight dependence of this nanoperiodic pattern by using repeated multiline scanning on the same area of PS surfaces, reported that repeated scanning changed the morphology of this patterned structure and that the change depended on the molecular weight. Later, Aoike et al. [33] results confirmed the Meyers et al. observations and revealed that the morphology of scratched surfaces of monodisperse PS films is dependent on the molecular weight. Moreover, recent works revealed the major role of molecular weight in friction and wear of polystyrene [34].

In the experimental work presented here, the obtained nanotribological results are discussed from the viewpoint of a relation between the nanofriction force and the macromolecular weight. Thus, the evolution of the friction force between the AFM tip and four amorphous atactic polystyrenes (PS-26, PS-118, PS-245 and PS-385) surfaces is discussed as a function of sliding velocity and applied normal force.

2. Experimental section

2.1. Materials

Polystyrenes used in this study, were made by anionic polymerization. Anionic polymerization details were given in a previous paper [35]. PS molecular weights (M_w) and polydispersity (M_w/M_n) values determined by GPC are listed in Table 1.

2.2. Surfaces preparation

Spin-coating is the most widely used technique for organic coatings based on polymer solutions. The obtained thicknesses generally range from 1 μm to 200 μm , but

thinner films can be produced with dilute solutions and high spinning rates. The homogeneity of the spin-coated film strongly depends on the quality of the pristine solution and the chosen combination of solvent, spinning rate and atmosphere. Solutions of PS/toluene were prepared in concentrations of 10 wt%. The samples were spun in open air for 60 s at 1000 rpm onto cleaned silicon (100) wafers. As spin-coating progressed and the liquid layer thicknesses became of the order of 1 μm or less, a progression of time-varying interference colors was seen. Since the color changes stopped within approximately 20 s of spin-up for all concentrations, it was inferred that a spinning time of 60 s was adequate for providing the minimum final film thickness. Films were allowed to dry in air, and then placed in a vacuum oven at 140 $^{\circ}\text{C}$ (above PS glass transition temperature) for 1 h, for removing residual toluene [36]. The film thickness was verified by the scratch test and was determined by AFM to be 0.5 μm (± 50 nm) for all spin-cast polystyrenes.

2.3. Gel-permeation chromatography (GPC) measurements

Gel-permeation chromatography was used in this study to accurately determine the relative molecular weights and molecular mass distribution of the polystyrene samples. Measurements were made using a Waters Associates (Model 510) Gel-Permeation Chromatograph operating at 30 $^{\circ}\text{C}$. Three columns were used in series packed with styragel beads. The solvent used was tetrahydrofuran (THF). The PS molecular weights were calculated by calibration with eight different PS standards with molecular weights between 1200 g mol^{-1} and 970,000 g mol^{-1} .

2.4. Contact angles measurements and surface free energy

An automatized Krüss G2 goniometer employing the sessile drop technique was used for equilibrium contact angles, θ , measurements. This instrument is equipped with automatic image acquisition and contact angle computation software. Water, formamide and α -bromonaphthalene were used as liquids to obtain corresponding contact angles values on both sides of the drop image. This process was repeated five times. Measurements were carried out in open air at 22 ± 2 $^{\circ}\text{C}$ temperature and $35 \pm 5\%$ relative humidity. Droplets volume was equal to 10 μl .

The equilibrium contact angle, θ , for a liquid drop on an ideal, homogeneous, planar, and rigid surface is related to the various interfacial tensions by the Young's equation given by

$$\gamma_{lv} \cos\theta = \gamma_{sv} - \gamma_{sl} \quad (1)$$

where γ_{lv} is the surface tension of the liquid in equilibrium with its saturated vapor, γ_{sv} surface tension of the solid in equilibrium with the saturated vapor of the liquid, and γ_{sl} is the interfacial tension between the solid and the liquid.

The Fowkes [37,38] method improved by Owens and Wendt [39] based on the contact angles of a series of test liquids can be combined with Young's equation Eq. (1) to give

Table 1

Average molecular weights (g mol^{-1}) derived from gel-permeation chromatography and polydispersity index.

PS name	M_w	M_n	M_w/M_n
PS-26	25940	23478	1.11
PS-118	118098	106882	1.10
PS-245	245326	219016	1.12
PS-385	384985	354690	1.09

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