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Investigation of nano-scale scratch and stick-slip behaviors of polycarbonate using atomic force microscopy



Jie Liu, Han Jiang*, Qian Cheng, Chaoming Wang

Applied Mechanics and Structure Safety Key Laboratory of Sichuan Province, School of Mechanics and Engineering, Southwest Jiaotong University, Chengdu, 610031, China

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Keywords: Nano-scratch Nano-friction Stick-slip Atomic force microscopy	To investigate the nano-scale scratch and friction behaviors of polycarbonate (PC), atomic force microscopy (AFM) was utilized to conduct the nano-scratch tests under different normal loads with a probe of 20 nm tip radius. Then a probe of 1 nm tip radius was employed for the scanning of scratched surface morphology. The stick-slip phenomena are found for all studied normal load levels. The nano-scratch deformation at different normal loads can be categorized into three modes: sliding on asperities, elastic deformed groove and permanent scratch groove. After the analysis of amplitude-frequency characteristics of friction force, a good correlation is found among scratch deformation, friction force and stick-slip. Those findings can be a good guidance to un- derstand and improve the nano-scratch performance of polymers.

1. Introduction

Polycarbonate (PC), as an amorphous glassy polymer, has been widely used in medical, electronic and auto industries due to its good optical properties, high strength and impact resistance. While the nanoscale surface scratch damage is not acceptable for the micro-electromechanical system (MEMS), the nano-scratch itself is an intuitive technology for fabricating nano-pattern on polymer surface to achieve certain surface characteristics. For both application scenarios, the understanding of nano-scratch performance of PC is necessary.

Because of its inherent viscoelastic plastic behaviors, polymer's scratch is a complex mechanical procedure. Researchers have studied the macro-scale polymer scratch behavior using experimental method [1-5] and finite element method [6-10]. The scratch damage mechanisms at macro-scale were discussed [4,5,8,10]. Those research efforts laid a solid foundation for understanding and improving polymer scratch performance. However, due to the intrinsic dissimilarity between the surface layer and substrate of polymeric materials, as well as the complicate surface effects, the nano-scratch behaviors could be quit different from those at macro-scale. Recently, researchers have paid attention to the nano-scratch behavior of polymer [11-16]. Mainly focusing on the nano-scratch deformation and friction behavior during the scratch, the experiment equipment such as atomic force microscopy (AFM) [13,16,17], nano-indenter [11] and nano-scratcher [15] have been utilized to investigate the effect of material mechanical properties, load conditions and surface characters on the nano-scratch performance

of polymeric materials.

Adams et al. [11] investigated the scratch damage of PMMA at a constant normal load of 100 mN by nano-indenter. The pile-up height of scratch groove was observed, and the strain hardening and elastic recovery of polymer material after scratch was demonstrated. Wong et al. [15] used nano-scratcher to study the effect of indenter geometry and material properties (e.g. surface roughness, ductility) on nano-scratch behaviors such as scratch depth, elastic recovery and scratch morphology of PC and PMMA at nanoscale. They found that there is a rubbery and ductile surface layer with a thickness of about 40 nm, whose physical and mechanical properties are different from those of bulk substrate. The nano-indentation hardness and nano-scratch hardness of ZnO/PMMA composites were measured by Chakraborty et al. [18]. The correlation between coefficient of friction (COF) and the scratch depth has been discussed. The nano-scale scratch characteristics of polymer materials have also been explored by Karimzadeh et al. [19,20], Noh and Fereidoon et al. [21]. AFM, as an extremely important tool to study the nano-scale material properties [19,22,23], has been used by Geng and Yanquan et al. [13,16,17] to investigate the effect of the normal load and scratch rate on the nano-friction behavior of PC and PMMA.

Although the relative motion between scratch tip and material surface is generally prescribed to be smooth, the stick-slip phenomenon during the scratch has been commonly observed from macro-scale to nano-scale, down to atomic-scale [24-28]. This phenomenon was discovered by Bowden and Leben for metallic materials [29]. Other

^{*} Corresponding author.

E-mail address: jianghan@home.swjtu.edu.cn (H. Jiang).

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Fig. 1. Scratched surface morphology: (a) Surface morphologies at different normal loads; (b) Typical longitudinal section profile of the scratch grooves at normal loads of 200 nN, 250 nN, 375 nN, 500 nN, 750 nN.

researchers also observed the stick-slip during polymer scratch [7,25,26,30]. The correlation between friction and stick-slip has been noticed by Zhang [31]. Zhang and Li et al. [30,31] investigated the effects of normal load and driving speed on the friction force of a styrene-acrylonitrile copolymer (SAN).

While great research contribution has been made on the field of polymer nano-scratch, few literature can been found for the evolution of various nano-scale scratch/friction modes and their corresponding mechanisms, not even mentioning the nano-scale stick-slip at all.

In this paper, AFM is employed to conduct the nano-scratch tests of PC under different normal load levels. To study the effect of surface characters and material mechanical properties on scratch behaviors, the after-scratch surface morphologies are scanned by AFM. The various scratch deformation modes and their mechanistic mechanisms are analyzed. The correlation between the amplitude-frequency character-istics of nano-scale scratch friction and the stick-slip phenomenon is demonstrated.

2. Experiments

2.1. Equipment and materials

An atomic force microscope with Nanoscope-V controller (Bruker AFM, MultiMode 8) was used to conduct nano-scratch and surface morphology scanning. Two types of probes with 20 nm and 1 nm tip radiuses were used for nano-scratch and surface scanning respectively. All tests were performed at a temperature of 24–28 °C and the relative humidity of 50%–80%.

The polycarbonate (PC, LEXAN PC0703, SABIC Innovative PlasticsTM) samples used for the tests were cut into the size of 10 mm*10 mm with a 5 mm thickness. Before scratch, the specimens

were ultrasonically cleaned with anhydrous ethanol for 10 min at room temperature and then air-dried. All specimens were glued on the magnetic iron sample holder.

2.2. Nano-scratch and surface characterization

Hardened diamond-like carbon (DLC) coated silicon probes (Series HARD, MikroMasch, CA, USA) with pyramidal shape tip were used for nano-scratch tests. The typical tip radius was 20 nm, thus the tip head can be taken as spherical when the scratch depth is less than 20 nm. The backside of the rectangular n-type silicon cantilever, coated with the 30 nm aluminum (Al) reflective film, owns a spring constant of 40 N/m and a typical resonance frequency of 325 kHz. Deflection sensitivity and lateral deflection sensitivity were calibrated on a sapphire surface (SAPPHIRE-12M, Bruker, CA, USA) and a patterned surface (VGRP-15M, Bruker, CA, USA), respectively. All nano-scratch tests were conducted in contact mode in air using AFM. The friction forces during scratch can be measured after the torsion spring constant of probes has been calibrated.

Various constant scratch normal loads, 1 nN, 10 nN, 25 nN, 50 nN, 75 nN, 100 nN, 150 nN, 200 nN, 250 nN, 375 nN, 500 nN, 625 nN, 750 nN, were applied respectively. The scratch speed was $2 \,\mu$ m/s, and the scratch length was $5 \,\mu$ m.

High resolution silicon probes (Hi'Res-C, MikroMasch, USA) with spike like tip were used to measure the surface morphologies at least 30 min after the scratch tests. The spike tip radius was 1 nm. The rectangular n-type silicon cantilever with a spring constant of 2 N/m and a typical resonance frequency of 75 kHz film is soft enough and no additional damage occurs on the sample surface during the high-resolution surface morphology scanning. All images were processed with the NanoScope Analysis (Bruker, CA, USA).

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