Structure and Wear Properties of Nano-silicon dioxide modified Polyacrylate composites

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Abstract: polyacrylate/nano-silicon dioxide nanocomposites were fabricated through a simple mixing process for resisting scratch and wear as coating. Chemical compatibility between polyacrylate matrix and nano-silicon dioxide (nano-SiO_2) was achieved by functionalized nano-SiO_2 via three silane coupling agents. Scanning electron microscopy (SEM) was done to characterize surface and wear morphology of the nanocomposites. Differential scanning calorimetry (DSC) was used for representing interfacial properties of the nanocomposites. A MM-200 machine and a Norman Tool RCA abrader were performed for testing friction and wear properties. SEM showed that incorporation of the nano-SiO_2 functionalized with silane into the polyacrylate matrix showed a better dispersion than the composite without silane at low nanoparticle content ($\leq$3 wt%). SEM also revealed that adhesive wear mechanism of the polyacrylate matrix was transited to dominated particle wear of the nanocomposites. An increase of glass transition temperature (T_g) was recorded via DSC at low nanoparticle content (1 wt%). Further addition of the nanoparticles to 3 wt% led to a 10°C increase in T_g comparing to neat polyacrylate. Results of friction and wear properties exhibited that friction coefficient and wear loss of the nanocomposites with silane were lower than those of the neat polyacrylate. The wear loss of the neat polyacrylate, the nanocomposite containing 3 wt% nanoparticles with silane KH570, and the nanocomposite with raw nano-particles were 108.6, 65.8, and 110.5mg, respectively. RCA results also showed a significant improvement of the nanocomposites in the presence of the nanoparticles with silane.

Introduction

Polyacrylate resin is a class of polymers that are known for their stability of resisting sunlight and temperature, favorable adhesive properties with basic materials and chemical resistance, which has been found applications in coating industries. Silicon dioxide is one of the most important materials for many fields such as chemical sensors, varistors, thin film photovoltaic solar cells, catalysts, and cosmetic meterials. So far, nano-coatings of acrylic resin or modified acrylic resin have been widely used in many fields such as building, polymer surface decoration, and so on.

The physical and mechanical properties of polymer/nanofiller composites are known to be strongly dependent on dispersion of fillers in polymer matrices. Many efforts including chemical purification and functionalization of the nanofillers[1], in-situ polymerization [2-3], and mechanical ball milling[4] have been put in force to disperse nanofillers more uniformly within the composites.

To estimate the amount of interfacial material and properties created by the introduction of nanoscale fillers, we processed and characterized a polyacrylate matrix nanocomposites with nano-SiO_2. Three types of silane were used as coupling agent for improving compatibility and interfacial interaction between the matrix and the nanofillers. DSC experiments were used to

understand the impact of the nanofillers on thermal transition and the shift of T_g indicated the effects by adding these nanofillers because of the amount of interfacial zone. The results also showed that friction and wear properties improved significantly in the presence of the nanofillers.

Experiments

Methyl methacrylate monomer (MMA), methyl butyl acrylate monomer (MBA), and azobisisobutyronitrile (AIBN) used in this research were purchased from Shanghai Sinopharm Chemical Reagent Co. Ltd, China. Silane (KH550, KH560, KH570) were purchased from Nanjing Shuguang Chemical Group Co., Ltd. Nano-silicon dioxide (nano-SiO_2, 20 nm) were purchased from Xiamen Lujiali Nanomaterials, Ltd.

MMA (43.5g), MBA (8.5g), AIBN (1g), and toluene (50g) were charged to a flask equipped with thermometer and stirring and refluxed for 4 hrs. Raw nano-SiO_2 were first sonicated for 10 min at 40 ºC in alcohol solution. Added 11 wt% silane of nano-SiO_2 content into former nano-SiO_2 alcohol solution, adjusting pH value at 3~4 with glacial acetic acid. The mixture was stirred at 75 ºC for 3 hrs to yield functionalized nano-SiO_2. Preweighted nano-SiO_2 were added into polyacrylate toluene solution with thoroughly stirring. Thereafter, the mixture was heated at 100ºC for 4 hrs, then degassed in a cacuum oven at 100ºC for 2 hrs, and heated at 120ºC for 4 hrs for curing.

The dispersion achieved of the nanocomposites was observed with a XL30-ESEM scanning electron microscope. All samples were sputter coated with gold prior to imaging. DSC experiments were conducted using a TA Instruments DSC 204. A Norman Tool RCA 7-IBB abrader was used for determining abrasion resistance of coating membrane. Friction and wear properties were tested by a MM-200 machine. The wear test was keeping for one hour, and applied load was 10 kg.

Results and discussion

Morphology of materials surface and wear surface of the nanocomposites

The material surface morphologies of raw nano--SiO_2, silane-functionalized nano-SiO_2 with diffirent nano-particle concentration are shown in Fig. l. There are some micropores and nano-particle aggregations on the surface of the composite with raw nano-particle in Fig. 1a. The composite with same concentration nano-particle functionalized by silane KH570 showed less micropores and better dispersion in Fig. 1b, which due to improved compatibility between the polyacrylate matrix and the nano-SiO_2 for the presence of KH570. However, the effect of the silane on the compatibility was restricted at higher nano-particle concentration, for instance, 5 wt%, which exhibited aggregations and irregular surface in Fig. 1c.
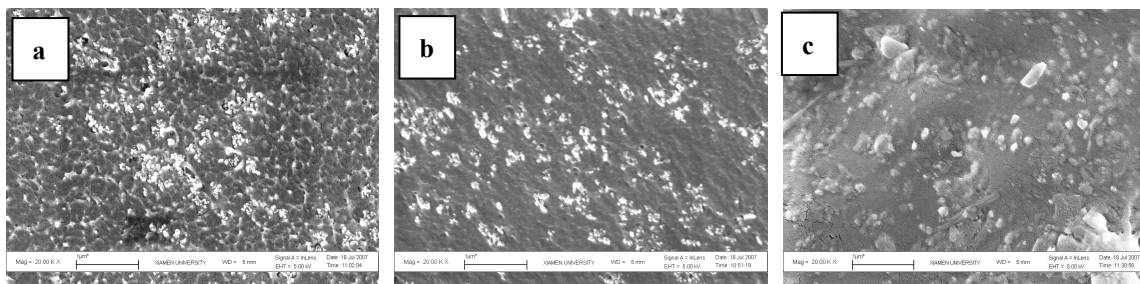

Fig. 1 SEM pictures of the nanocomposites. (a) raw nano-SiO_2 (3 wt%), (b) KH570 functionalized nano-SiO_2 (3 wt%), and (c) KH570 functionalized nano-SiO_2 (5 wt%)

The neat polyacrylate indicated a dominative adhesive wear, going with tearing resin in Fig.2. All nanocomposites with 3 wt% nano-particles exhibited stronger ability for resisting the friction load, showed soft adhesive wear on the wear surfaces of the nanocomoposites. The composite containing 3 wt% raw nano-particles showed split on the surface due to weak interfacial interaction.

The composites containing 3 wt% nano-particles with silane exhibited stronger interaction between the matrix and the nano-particles, which led to increased resistance to the friction.
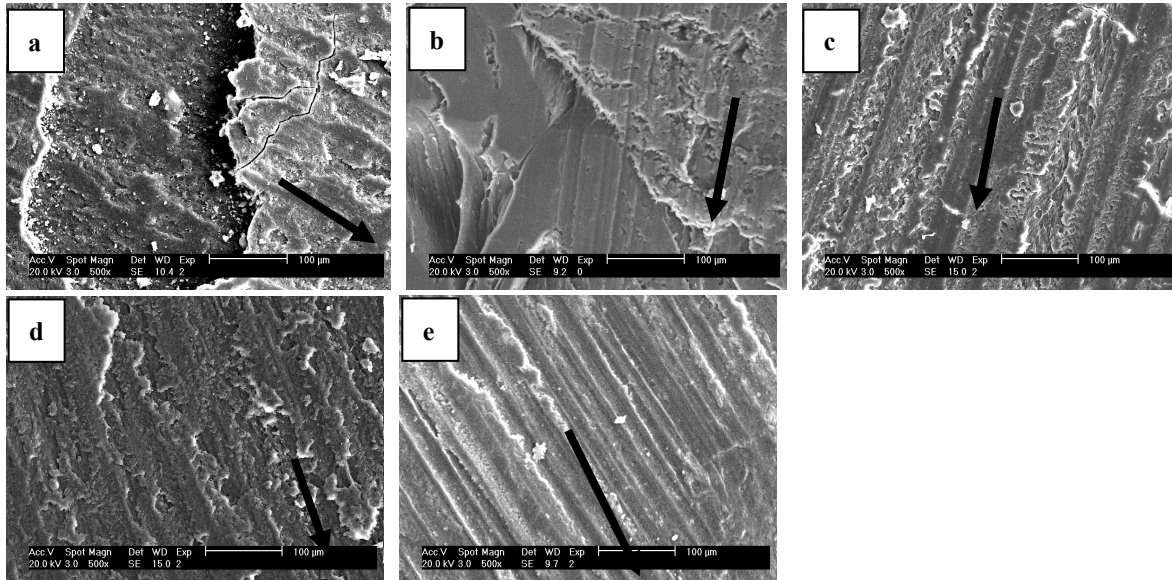

Fig.2 Wear surface SEM pictures of the nanocomposites with 3 wt% nano-particles. (a) polyacrylate matrix, (b) raw nano-particles, (c) silane KH550, (d) silane KH560, (e) silane KH570)

Effects of coupling agents on the friction and wear properties of the nanocomposites

Results in Table 1 exhibited that the coefficients of the nanocomposites with silane such as KH550, KH560, and KH570 were lower than the neat polyacrylate and the composite with raw nano-particles. This difference was attributed to the silane, which improved the compatibility and interfacial interaction between the matrix and the nano-SiO_2. Table 1 also showed that the wear loss with KH570 was decreased from 108.6 mg to 65.8 mg, this enhancement was attributed to the effective interaction between the nanofillers and the matrix that were covalently-bonded with the nano-particles. Compared to silane KH550 and KH560, there were similar improvement on the nanocomposites. Similar phenomenon has not been found in the nanocomposite with raw nano-particles. Table 1 also indicated that wear loss decreased significantly by incorporation the nano-SiO_2 at $\leq$ 3 wt%. The nanocomposites with more than 4 wt% nano-particles showed a soft increase in wear loss due to aggregations and poor dispersion.

Table 1 Friction and wear properties of the nanocomposites

properties	Nanofiller content (wt%)	Friction coefficient	Wear loss (mg)	Wear rate (%)
Polyacrylate matrix	/	0.48	108.6	7.18
Raw nanofillers	3	0.55	110.5	7.31
KH550	3	0.36	66.1	4.37
KH560	3	0.35	65.5	4.33
KH570	1	0.45	83.3	5.51
	2	0.44	75.5	4.99
	3	0.32	65.8	4.35
	4	0.40	70.3	4.65
	5	0.53	71.0	4.70

DSC results in Table 2 suggested that the T_g increased with the nano-particle content increase at a small amount ($\leq$3 wt%). For instance, the T_g of the nanocomposite containing 3 wt% nano-SiO_2 was 10 °C higher than the matrix. It means nano-particle plays a significant role in restricting

movement of the macromolecule chain, indicating that introduction of the nano-particles functionalized with silane KH570 enhanced the interfacial interaction between the matrix and the nano-fillers. However, the T_g of the composite with higher amount (5 wt%) of nano-particles decreased due to increased aggregations, which was shown in SEM micrograph in Fig. 1.

RCA test is a common wear testing method for characterizing coating wear of products' surface, such as laptop, mobile phone, and so on. The RCA times tended to increase when nanoparticle content was ≤3 wt%. The hardness of the coating as shown in Table 2 increased as the increase of the inorganic nano-SiO$_2$, at the same time, resulted in RCA improvement.

Table 2 Properties of the polyacrylate/nano-SiO$_2$ composites

Nano-SiO$_2$ content (wt%)	0	1	2	3	4	5
T_g (°C)	87.3	90.7	/	97.4	/	92.9
Hardness	HB	HB	HB	H	H	2H
RCA test (times)	5	8	8	13	11	10

Summary

Polyacrylate matrix nanocomposites filled with nano-SiO$_2$ were fabricated by a simple mixing process. Three silanes were used to improve the compatibility and interfacial interaction between the matrix and the nanofillers. The effects of the nanofillers on the dispersion, morphology, and friction and wear behavior of the nanocomposites were studied. The results of SEM indicated that the nanofillers with silane exhibited much better dispersion than the raw nanofillers. The morphology of wear surface also suggested that the nanocomposites with functionalized nanofillers showed an increased ability for resisting friction load, due to improved interfacial interaction for silane coupling agents. The increased T_g also testified the results. Friction and wear results indicated that the nanocomposites with 3 wt% functionalized nanofillers showed lower friction coefficients than the matrix and the nanocompsite with raw nanofillers. Furthermore, the wear loss of the nanocomposites showed significant decrease by incorporation the functionalized nanofillers. The wear loss of the polyacrylate matrix, the nanocomposite containing 3 wt% nanoparticles with KH570, and the nanocomposite with raw nanofillers were 108.6, 65.8, and110.5 mg, respectively. RCA   results showed that the nanocomposite with 3 wt% nanofillers increased from 5 times of the neat polyacrylate to 13 times.

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