



Wear-resistant and transparent acrylate-based coating with highly filled nanosilica particles

Hui Zhang^a, Hui Zhang^a, Longcheng Tang^a, Zhong Zhang^{a,*}, Lei Gu^b, Youzhong Xu^b, Christian Eger^c

^a National Center for Nanoscience and Technology, 100190 Beijing, China

^b Chery Automobile Co. Ltd., 241009 Wuhu, Anhui Province, China

^c Nanoresins AG, 21502 Geesthacht, Germany

ARTICLE INFO

Article history:

Received 3 December 2008

Received in revised form

25 April 2009

Accepted 1 May 2009

Available online 28 May 2009

Keywords:

Silica nanoparticles

Nanocomposites

Hybrid coating

Wear resistance

ABSTRACT

Hybrid nanocoatings are one of the most attractive topics in nanomaterials which have achieved the transition from fundamental researches to practical applications. In the present study, a urethane–acrylate oligomer was mixed with varied concentrations of nanosilica particle sol, spin-coated onto polycarbonate substrate and finally cured by ultraviolet (UV) rays. The morphology, mechanical properties and wear resistance of the resultant hybrid coatings were systematically investigated. Infrared spectroscopy (IR) analysis was performed to determine the eventual curing extent of the mixtures studied. The transmission electron microscopy (TEM) micrographs revealed almost perfect dispersion of the nanosilica particles within organic matrices, which ensured the excellent transparency of the hybrid coatings. Nanoindentation was further conducted to determine the mechanical properties, i.e. hardness, elastic modulus and their nanoparticle loading dependence. The short-term wear resistance was characterized by a pencil hardness tester. Moreover a universal micro-tribotester (UMT) was applied to investigate the long-term performance. As a result, about 20% decrease in coefficient of friction (COF) was achieved by the coating filled with 40 wt% nanosilica particles, compared to that of the unfilled coating. Under the same fretting test conditions, the wear rate in terms of wear volume of the hybrid coating containing 40 wt% nanoparticles was about 70 times lower than that of the neat coating, confirming the wear-reduction capability of the nanoparticles. The related wear mechanisms were discussed based on worn-surface observations.

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1. Introduction

Hybrid nanocoatings are one of the most attractive topics in nanomaterials which have achieved the transition from fundamental researches to practical applications. In recent years, organic/inorganic hybrid nanocoatings have been successfully used in a wide range of applications [1–4], especially on polycarbonate (PC) which have several advantages over the use of conventional transparent materials (glasses, PMMA). These transparent hybrid coatings are usually formed by introducing inorganic nanoparticles to polymeric matrices, of which acrylates and their derivatives are the mostly used since they exhibit excellent optical properties and can be conveniently polymerized with UV rays. UV-curing has become a well-accepted technology due to its unique advantages [5].

The nanoparticles are under consideration in hybrid coatings since they can give special hints to improve the hardness, elastic

modulus and wear resistance of host polymeric coatings without losing their transparency once nanoparticles are homogeneously distributed in the polymer matrices [4,6–9]. However, it is well recognized that a basic challenge of nanocoating processing is the dispersion technique. Due to their strong adhesive force, nanoparticles tend to stick together so as to form agglomerates or clusters in matrices, consequently resulting in mechanical property and transparency degradation. Conventional processing approaches such as mechanical mixing and melt compounding are usually difficult to achieve uniform particle distribution at the nanoscale [10]. An effective method for obtaining these agglomerate-free hybrid coatings is the *in situ* sol–gel technique, which introduces different content of nanoparticles into monomers or oligomers by chemical reactions [4,11–13].

In polymeric coatings, nanoparticles have been used to upgrade the level of resistance to wear and friction. Apart from the mostly used nanosilica particles, other nanoparticles were also chosen to improve the wear resistance of polymeric resins, such as nanolumina, nanotitania and nanozirconia et al. [9]. Such improvements are usually ascribed to the strong interfacial interactions between organic and inorganic phases. For example, the interphase around

* Corresponding author.

E-mail address: zhong.zhang@nanoctr.cn (Z. Zhang).

nanoparticles was reported to become harder than the bulk polymer matrix [4,11]. Moreover, chemical groups on the surface of nanoparticles could result in more covalent linkage between particle and matrix [14]. An interesting finding is that nanoparticles, combining with a proper amount of micron-sized particles, can further improve the wear resistance of polymeric coating, which was superior to that of materials filled with nanoparticles or micron-sized particles only [7,8,15]. In addition, nanoparticles can increase the glass transition temperatures (T_g) of some polymer systems [12,16]. From the point of view of long-term wear process, the increased T_g allows the nanocomposites to endure much friction heat during wear and to have better wear resistance, as compared to the host polymers without nanoparticles.

With respect to wear mechanisms of nanoparticles, they involve, as proposed by other researchers, the enhanced mechanical performance (e.g. modulus, hardness and fracture toughness), the rolling effect between material pairs or the improved bonding between transfer film and metallic counterpart [17–20]. Zhang et al. [21] reported that the limiting pv values (the product of the normal pressure p and the sliding velocity v) of epoxy resin can be remarkably enhanced after addition of nanoTiO₂ particles because of the nanoparticle-induced positive rolling effect between material pairs. Such an effect was also found by Rapoport et al. [22,23] when WS₂ nanoparticles with close-caged structure were used in the lubricant oil. Moreover the nanoparticles could also enhance the cohesion strength of transfer film to counterparts. The nanoTiO₂ particles contribute to an improved wear resistance by changing from a severe abrasive wear of the neat epoxy to a mild abrasive wear caused by formation of a compacted transfer film on the counterpart [24]. Bahadur et al. [25,26] found polyphenylene sulfide (PPS) filled with different kinds and amounts of nanoparticles exhibited stronger bond strengths and lower wear rates owing to the ability of the nanoparticles to ‘anchor’ transfer films to counterfaces. Wang et al. [27–30] showed that the addition of nanoparticles, such as Si₃N₄, SiO₂, SiC and ZrO₂, could effectively reduce the coefficient of friction (COF) and wear rate for PEEK-based nanocomposites. The dominant mechanisms were modified from an adhesive and fatigue wear of the neat PEEK to a mild abrasive wear of the nanocomposites as a thin, uniform and tenacious transfer films were formed.

As mentioned above, nanoparticles play an essential role in improving the wear-resistance of polymer coatings. Although there are lots of investigations on hybrid nanocoatings in the literature [4,11,14,15,31,32], the nanoparticle contents in those works are relatively low, this is mainly due to the difficulty in dispersing and preparing high-concentration nano/polymer coatings. In this work we successfully prepared acrylate-based coatings filled with high concentration of silica nanoparticles without any agglomerates, and their mechanical properties, wear resistance and optical properties were systematically studied. Although the concentration of nanosilica reached up to 40 wt% in this work, the coatings were almost completely transparent. Both the short-term and long-term wear resistance was characterized by a pencil hardness tester and a UMT tester, respectively. It is noted that the fretting

resistance (in terms of wear volume) was dramatically improved and a minimum COF value was obtained when embedded nanosilica particle content was 40 wt%. Moreover, this study was also focused on the analysis of the wear processes and friction behaviors of the nanosilica-filled coatings. The related wear-reductive mechanisms were discussed based on the morphologies of the worn surfaces of nanocoatings and their counterparts.

2. Experimental

2.1. Materials

A commercially available hexa-functional aliphatic urethane–acrylate (UA) oligomer and trimethylolpropane triacrylate (TMPTA) that was a commonly used reactive diluent with three functional groups were supplied by Cytec Industries Inc. A colloidal nanosilica sol with a trademark ‘C150’ was kindly supplied by nanoresins AG, Germany. It consisted of about 50 wt% sol–gel-formed silica nanoparticles and 50 wt% TMPTA serving as the solvent. A radical photoinitiator (1-hydroxycyclohexyl phenyl ketone, Irgacure 184) was obtained from Ciba Specialty Chemicals. All these chemicals were used without further purification.

2.2. Coating preparation

The UV-curable coatings were prepared as follows. The colloidal nanosilica sol was mixed with different amounts of UA, TMPTA and Irgacure 184, and then the mixtures were intensively stirred at room temperature until clear dispersions were obtained. After degassing under reduced pressure, a proper amount of dispersion was dropped onto a polycarbonate plate (1 mm in thickness) and spin-coated at a rotation speed of 2000–4000 rpm. The wet films were subsequently UV-cured at room temperature for 180 s using a hot embossing system HEX01 equipped with UV unit (JENOPTIK Mikrotechnik GmbH), which gave the UV light intensity of 1.5 mW/cm² at a wavelength of 365 nm. The curing process proceeded under reduced air pressure of 4 mbar in order to avoid the polymerization inhibition of oxygen. The thickness of resultant solid coatings depended mainly on the processing conditions and dispersion viscosity and can be controlled in the range of 20–30 μm, which accurately measured using a surface profilometer (Dektak 150, Veeco, USA). In the final coating formulations, the weight ratio of UA to TMPTA maintained constant (UA/TMPTA = $\frac{1}{2}$), a photoinitiator of Irgacure 184 was introduced to the compositions of UA and TMPTA at concentration of 4 wt%, only the silica nanoparticles loadings varied from 10 to 40 wt%. The detailed formulation of the coatings prepared is summarized in Table 1.

2.3. Characterization

Fourier-transform infrared (FT-IR) spectroscopy (Spectrum One, Perkin-Elmer Inc., USA) can be used for quantitative analysis

Table 1
Compositions of coating samples with various nanosilica particle content.

Coating sample	Theoretical silica content (wt%)	C-150 ^a (wt%)	UA ^b (wt%)	TMPTA ^b (wt%)	Irgacure 184 (wt%)
Neat coating	0	0	33.3	66.7	4
10 wt% Nanosio ₂	10	20.0	30.0	50.0	4
25 wt% NanoSiO ₂	25	50.0	25.0	25.0	4
40 wt% NanoSiO ₂	40	80.0	20.0	0	4

^a C-150 is a trademark of colloidal nanosilica sol containing about 50 wt% sol–gel-formed silica nanoparticles and 50 wt% TMPTA serving as the solvent.

^b In final coating formulations, the weight ratio of UA to TMPTA maintained constant (UA/TMPTA = $\frac{1}{2}$).

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