



Friction and wear properties of hybrid sol–gel nanocomposite coatings against steel: Influence of their intrinsic properties

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ABSTRACT

A dual UV-curing process inducing in a single step cationic photopolymerization and a photoinduced sol–gel process was used to obtain novel hybrid coatings. For this, an epoxy resin based on hydrogenated diglycidyl ether bisphenol A was mixed with an epoxy trimethoxysilane precursor (GPTMS, TRIMO) in the presence of photoacid generator based on diaryliodonium salt. Various UV-cured coatings were prepared with different amounts of the hybrid monomer (20 and 50 wt%), and two thicknesses: 15 and 80 μm . The friction and wear properties of these coatings were characterized on a ball-on-disc tribometer (steel ball; applied normal load: 6 N; sliding speed: 5 cm/s). Both the coating thickness and the addition of the hybrid monomer tend to improve the stiffness of the pure epoxy resin; however, these two parameters also induce an increase of the dynamic friction value.

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1. Introduction

Polymeric materials aim to become more and more important in various industries as attractive substitutes to metals. Many studies have been conducted on the friction and wear of polymers [1–3], and it appears that the two major components of friction are adhesion and deformation. However, much more deserves to be done to thoroughly study the behaviour of polymers against various counterfaces.

As previously mentioned, the most commonly observed process is adhesion, going together with a transfer of the polymer to the metal or ceramic counterface; here, both the friction coefficient and wear rate are high [4,5]. The friction can however be reduced if a softening of the surface occurs [6]. Fatigue wear is also quite important; this process can be considered as a thermally activated failure mechanism [7]. The formation of the wear debris can be initiated either in the surface or in the subsurface layer of the polymer [8]. These are only a few examples of the numerous processes that can occur in a contact including a polymer as one of the counterbodies.

Epoxy resins are widely used in many industrial applications. However, when sliding against a hard counterface, the presence of flaw formation, cracks nucleation and propagation in the surface layer of the polymer are evidence of fatigue process [9]. In addition, these materials exhibit a high friction coefficient. To remedy these drawbacks, many studies were carried out in order to modify the

epoxy resins with various additives. For example, a fluoroepoxy oligomer (diglycidyl of trifluoromethyl aniline) was mixed with a commercial diglycidylether of bisphenol A epoxy: the addition of tertiary amines leads to a decrease of the dynamic friction value and the formation of a protective transfer film tends to diminish the wear rate [10]. The use of fluorine functional groups to chemically modify the epoxy resins induces low friction, playing thus the role of solid lubricants. Therefore, it can either decrease the adhesion or create a protective film with low interfacial shear stress in the contact, that decreases the friction and wear [11].

Even in UV curing technology, different routes were investigated to change the epoxy properties. The current trend is to synthesize hybrid materials, obtained from the mixing of an epoxy resin and various hybrid monomers.

Most of the synthesis methods of this kind of coatings are based on a two-step process:

- nanoparticles are mixed with a cationically reactive resin; then the UV irradiation is performed to create a cross-linked hybrid system [12,13],
- metal alkoxide precursors are introduced in an epoxy or vinyl ether monomer; the photopolymerization can occur before or after the sol–gel reaction [14,15],
- a sol–gel reaction is first carried out, with a hybrid precursor, to obtain a liquid epoxy functional polysiloxane network; this latter is then photopolymerized [16,17].

However, recent studies on cationic photopolymerization have shown that the same kind of coatings can be synthesized in an only

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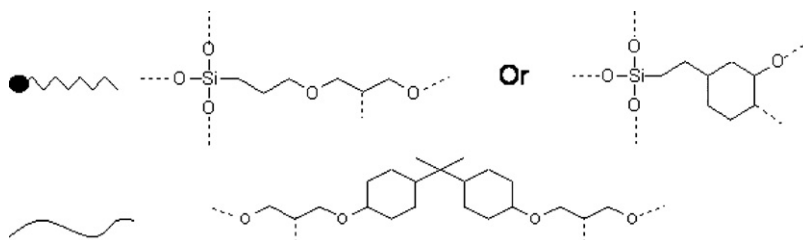


Fig. 1. Representation of the organic–inorganic cured network [18].

one-step simplified process; the so obtained films are moreover constituted of networks that are more homogeneous and better interpenetrated [18,19]. The present paper deals with an original one-step process to obtain the organic–inorganic coatings. A reactive organosilane (GPTMS or TRIMO) and a cationically UV-curable resin (Epalloy 100) are mixed and exposed to UV, in the ambient (20 °C, 30–40%RH), in the presence of a cationic photoinitiator which is able to generate Brønsted acids upon photolysis. Previous works on this method [20,21] have proved that the diffusion of water originating from the atmosphere is sufficient to induce the hydrolysis of the trialkoxysilyl functions in the presence of the photoinitiator. The photoacid-catalysed cross-linking of the trialkoxysilyl groups and the cationic photopolymerization of the epoxy functions occur simultaneously, leading thus to the formation of both a three dimensional Si–O–Si network and a polyether chains network, these two networks being closely linked. Moreover, the epoxy functions of the hybrid monomer can be copolymerized with the one of the cationic resin, inducing links between the organic and inorganic phases.

The structure of both organic and inorganic networks depends on the nature of the hybrid precursor incorporated in the formulation. The resulting coatings were extensively characterized by solid state NMR (Nuclear Magnetic Resonance) and FTIR (Fourier Transform Infra-Red) spectroscopies [21].

The nature and amount of the hybrid monomer, as well as the thickness of the coating, are intrinsic parameters of these materials whose influence on the friction and wear are studied here. In this work, the possibility to improve the tribological properties of conventional UV-curable resin by their copolymerization with hybrid-epoxy-alkoxysilane monomers was investigated.

2. Experimental

2.1. Synthesis of the organic–inorganic coatings

The films used in this study were obtained under UV-induced cationic polymerization; an hydrogenated diglycidyl ether bisphenol A (EPALLOY 5000, CVC Chemicals, used as received), named E100 in the following, was mixed with two different organoepoxysilanes (Aldrich, used as received):

- trialkoxy-silane 3-(glycidyloxypropyl)trimethoxysilane (GPTMS),
- [2-(3,4-epoxycyclohexyl)ethyl]trimethoxysilane (TRIMO).

The photoinitiator I250 (Ciba Specialty Chemicals) is a (4-methylphenyl)[4-(2-methylpropyl)phenyl] iodonium hexafluorophosphate. It is a 75 wt% solution of the active substance in propylene carbonate. The structure of the cationic photoinitiator and of the monomers are shown in Table 1.

Byk 333 (BYK Chemie) is a surface wetting agent based on a polyether modified polydimethylsiloxane.

2 wt% of I250 and 0.3 wt% of Byk 333 were added to the mixing of the organic-hybrid monomers.

Various formulations were obtained by using 20 and 50 wt% of GPTMS and TRIMO in EPALLOY 5000: afterwards, ExGy (or ExTy) will be used to identify a composite including x wt% of EPALLOY 5000 and y wt% of GPTMS (or y wt% of TRIMO).

The hybrid photocurable mixtures were spread on glass plates (washed with a 20 wt% NaOH solution, demineralised water and ethanol) and cured under an UV conveyor system (Qurtech equipped with a H-bulb lamp): the samples passed five times under the lamp, the speed of the belt was set at the constant value of 10 m/min, and the lamp intensity was 100%. The light dose received by the samples in these conditions was 7.3 J/cm² (UVA: 2.25 J/cm²; UVB: 2.1 J/cm²; UVC: 0.45 J/cm²; UVV: 2.5 J/cm²). The final epoxy conversion ranged between 95 and 100%.

Two coatings thicknesses were selected for this study. The thickness was controlled with an Altisurf 500 workstation (Altimet) equipped with a 350 μ m AltiProbe optical sensor. Thin and thick coatings (15 and 80 μ m respectively) were characterized in this study by changing the amount and the nature of the hybrid monomer introduced in the photopolymerizable mixture:

- thick coatings (80 μ m): E100, E80G20, E80T20,
- thin coatings (15 μ m): E100, E80G20, E80T20, E50G50, E50T50.

The final organic–inorganic cured network is shown in Fig. 1, as it was described in a previous paper [21].

2.2. Tribological measurements

The friction tests were carried out on a classical CSM ball-on-disc tribometer (Fig. 2), in the ambient (20 °C, 40%RH), as described

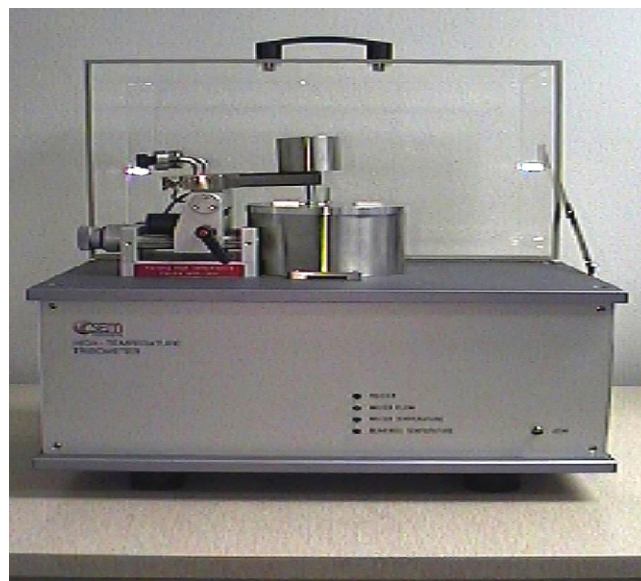


Fig. 2. CSM ball-on-disc tribometer.

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