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Modified epoxy coatings on mild steel: Tribology and surface energy

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ABSTRACT

A commercial epoxy diglycidylether of bisphenol-A (DGEBA) was modified by adding fluorinated poly(aryl ether ketone) fluoropolymer and in turn metal micro powders (Ni, Al, Zn, and Ag) and coated on mild steel. Two curing agents were used; triethylenetetramine (a low temperature curing agent) and hexamethylenediamine (a high temperature curing agent) for understanding the curing temperature effect on the properties. Variations in tribological properties (dynamic friction and wear) and surface energies with varying amounts of metal powders and curing agents were evaluated. When cured at 30 °C, dynamic friction and wear decrease significantly due to phase separation reaction being favored between the fluoropolymer and the epoxy. However, when cured at 80 °C, friction and wear increase; this can be explained in terms of a crosslinking reaction favored at that temperature. There is a significant decrease in surface energies with the addition of modifiers.

1. Introduction

Epoxies are noted for their versatility, high resistance to chemicals, outstanding adhesion to a variety of substrates, toughness, high electrical resistance, durability at high and low temperatures, low shrinkage upon cure, flexibility, and the ease with which they can be poured or cast without forming bubbles [1–5]. These properties make them eligible for use in various applications such as protective coatings (for appliance, automotive primers, pipes) [6], encapsulation of electrical and electronic devices, adhesives, bonding materials for dental uses, replacement of welding and riveting in aircraft and automobiles, composites materials in space industry, printed circuitry, pressure vessels and pipes, and construction uses such as flooring, paving, and airport runway repair [1,7].

One of the ways of improving the performance of polymers is by introducing a metallic dispersed phase. Metallic materials have useful properties and characteristics that are crucial for many applications; among them high electric conductivity, paramagnetism, high thermal conductivity as well as good mechanical properties. Combination of polymers with metals results in materials with electrical and magnetic properties comparable to neat metals and with a significant improvement in thermal properties of polymers. Also, the processability is the same as for neat polymers – a significant advantage for speed of production and processing costs.

Al particles added to poly(ethylene oxide) affect electric conductivity as reported by Muszynska and her colleagues [8]. Mamunya et al. used Cu and Ni powders as fillers in an epoxy resin and in poly(vinyl chloride) and studied the concentration dependence of electric and thermal conductivity [9]. Kim and coworkers added a soft Al + Fe + Si magnetic powder to a polymeric matrix to

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produce magnetic films to be used for shielding of electromagnetic waves [10]. Metal powders have been distributed in low density polyethylene (LDPE) without agglomeration [11], resulting in improvement of tribological properties [12,13]. Brito and Sanchez [14] used Zn, Cu, and Al as fillers in thermoset polymer systems composed by epoxy and amino resins at several concentrations up to 30 wt.%; they studied mechanical properties as well as thermal decomposition. They observed that the temperature of decomposition decreases when metal is added to epoxy + amine resins, this for all metals and all epoxy:amine ratios.

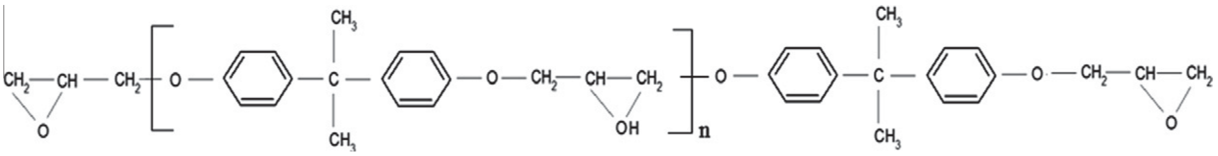
Modification of epoxies for achieving a wide range of properties including the above mentioned ones can be effective. Fluoropolymer modified epoxy provides better scratch resistance, low surface tension, better overall tribological properties and hydrophobicity [15–18]. Kumar, Alagar and Mohan developed siliconized epoxy interpenetrating coatings over mild steel wherein Zn powder was used for achieving good corrosion resistance [19]. Bazyliak, Bratychak, and one of us proposed the use of oligomers containing peroxy groups as epoxy modifiers in order to improve adhesion [20–22]. Polymer composites including carbon nanotubes (CNTs) are used for a variety of applications [23]; epoxies as corrosion protection coatings on steels have been reported [24]. Epoxy powder coatings modified using nano-$CaCO_3$, resulting in tensile and corrosion resistant properties were described by Yu and coworkers [25]. There has also been work reported on blends of thermoplastics like polysulfones and poly(methyl methacrylate) with epoxies for achieving toughness [26,27]. Kumar, Balakrishnan, Alagar and Denchev used silicone and phosphorus for modifying an epoxy to achieve anticorrosion, antifouling and flame retardant properties [28]. Fibers can also be used to alter the properties of the material [29].

In this work, modification of pure epoxy had been carried using a fluoropolymer and metal powders to achieve an advantageous combination of tribological and hydrophobic properties.

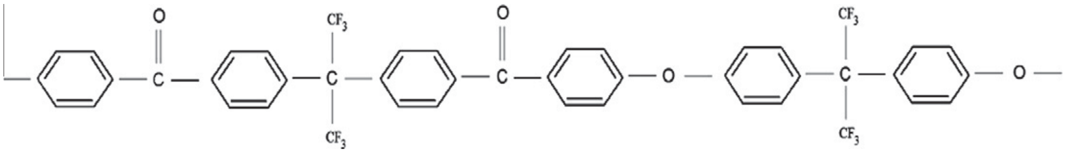
2. Experimental

2.1. Materials

Epon™ Resin 828, an undiluted clear difunctional bisphenol-A/epichlorohydrin derived liquid epoxy resin, diglycidyl ether of bisphenol-A (DGEBA), from Hexion Speciality Chemical Inc., was used as the base epoxy. The glass transition temperature T_g of the epoxy is 92 °C [16]. The epoxide equivalent weight of the epoxy is 185–192 g/eq. Its chemical formula is:



Fluorinated poly(aryl ether ketone) (FPEK) fluoropolymer was used as one of the modifiers due to its low surface energy nature which could provide hydrophobicity to the material and for its scratch resistance [19]. FPEK was synthesized in the Department of Chemistry, Texas State University, San Marcos according to a procedure reported in [30,31]. The T_g of the FPEK is 180 °C. Its chemical formula is:



The other modifier used was a metal powder. Four types of metal powders of size 1–5 μm were used: Ni, Al, Ag, and Zn. These metals powders were obtained from Atlantic Equipment Engineers (a division of Micron Metals Inc.). The Ni particles were used in flakes form whereas the Al particles were atomized particles. The shape of Ag was irregular, whereas the Zn particles were a combination of irregular and spherical particles.

Two types of curing agents were used in this work. One of them is triethylenetetramine (TETA) – a room temperature curing agent (from Hexion Speciality Chemical Inc.) and the other is hexamethylenediamine (HMDA) – a high temperature curing agent (from Sigma Aldrich). It has been reported in literature that curing temperature has a significant effect on a variety of properties – especially mechanical ones, friction, tensile bond strength, electrical resistivity, etc. of cured epoxies [16,32,33].

2.2. Epoxy modification, coating, and curing

Amount of FPEK used was 10 wt.% of the total system. Amount of curing agent used was 13 g TETA/100 g epoxy; 15 g HMDA/100 g epoxy. 25 wt.% metal powder/100 g epoxy was used.

Calculated amount of FPEK was dissolved in chloroform (20 ml chloroform/1 g FPEK) and then the epoxy resin was added. Then calculated amounts of metal powders were in turn added. This mixture was subjected to vacuum to remove chloroform and any trapped air. The curing agents were then added accordingly. These epoxy mixtures were coated onto ASTM A366 steel substrates (1″ × 2″) using a brush. Thus, two different types of systems were synthesized using TETA and HMDA. Each system consisted of six compositions:

Unmodified epoxy; epoxy + FPEK; epoxy + FPEK + Ni; epoxy + FPEK + Al; epoxy + FPEK + Ag; epoxy + FPEK + Zn.

Samples with TETA were cured at 30 °C (24 h), whereas samples with HMDA were cured at 30 °C (5 h) and then post-cured at 80 °C (24 h) to allow formation of a smooth and even coating. After curing all the samples were stored at room temperature.

2.3. Friction determination

Nanovea pin-on-disc tribometer from Micro Photonics, Inc., was used for determining dynamic friction. A SS 302 grade stainless steel ball with diameter 3.20 mm was used as the pin. The pin was loaded onto the test sample with a known weight 5.0 N. A highly stiff elastic arm insures a nearly fixed contact point and thus a stable position in the friction track. Dynamic friction is determined during the test by measuring the deflection of the elastic arm by

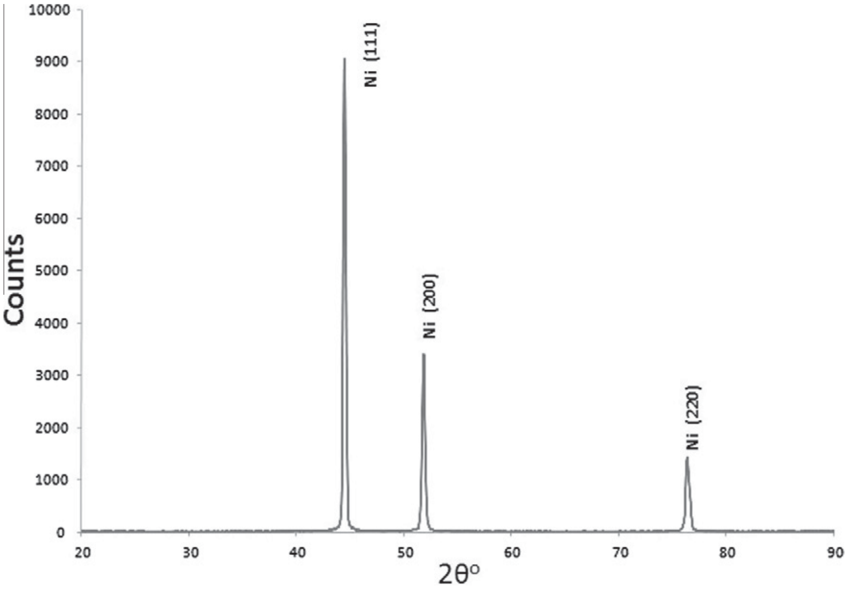


Fig. 1a. Diffraction pattern of Ni powder.

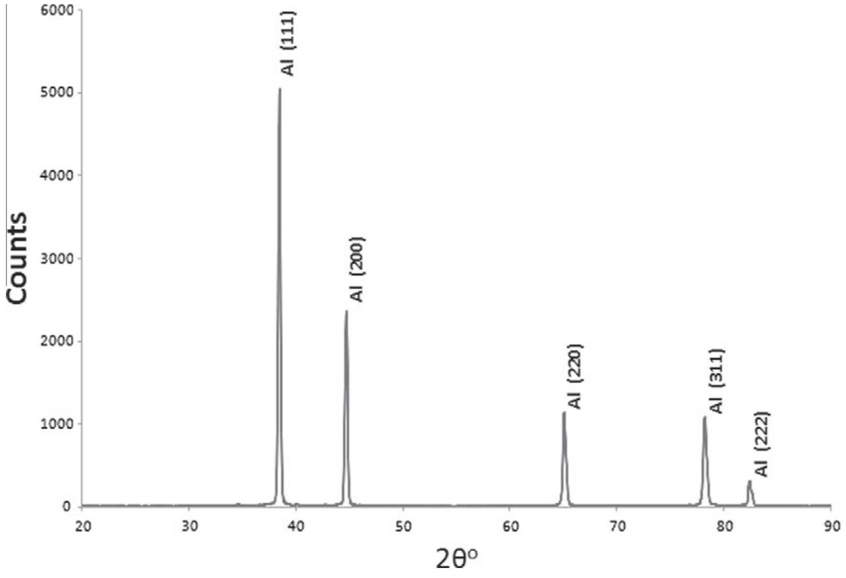


Fig. 1b. Diffraction pattern of Al powder.

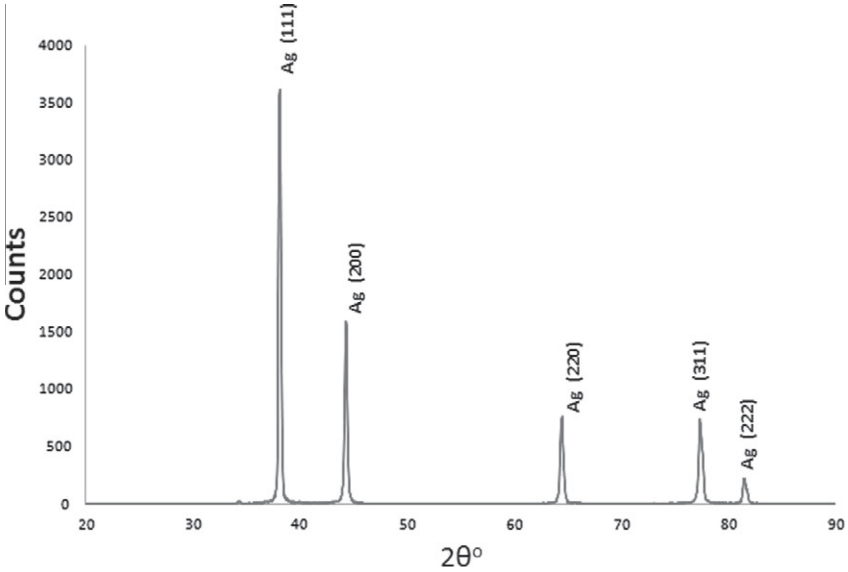


Fig. 1c. Diffraction pattern of Ag powder.

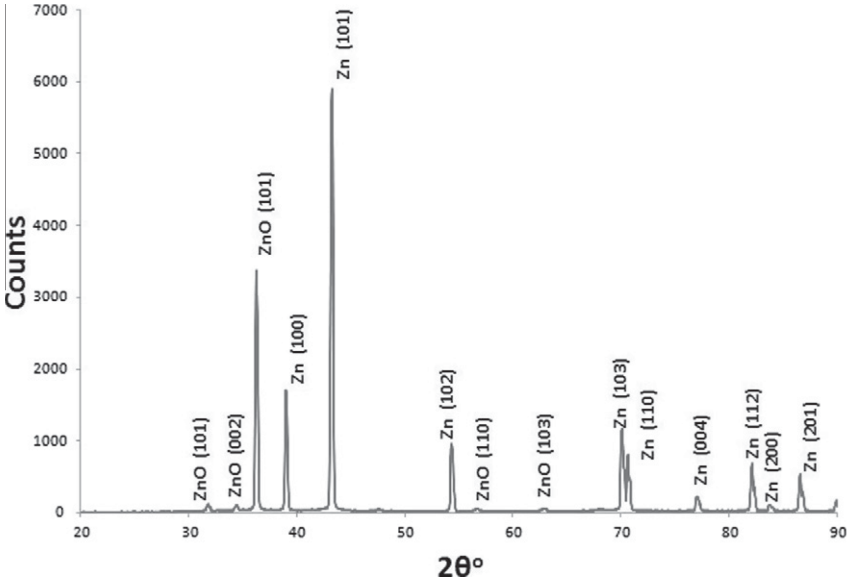


Fig. 1d. Diffraction pattern of Zn powder.

direct measurement of the change in torque. The rotation speed of the disc was 100 rpm and the radius of wear track was 2.0 mm. The test was performed for 500 and 5000 revolutions under room temperature conditions. The results reported are averages from three runs.

2.4. Wear rate determination

Wear was determined by studying the wear track resulted due to the pin-on-disc friction test after 5000 revolutions. FEI Quanta 200 SEM was used for analysing the wear track and determining the wear track width. The accelerating voltage was 20.0 kV. Ten values of wear track width were measured at different locations on each sample and averaged for the purpose of accuracy. The volume loss due to wear V_m was then calculated using the following formula according to ASTM G99-05:

$$V_m = 2\pi R[r^2 \sin^{-1}(d/2r) - (d/4)(4r^2 - d^2)^{1/2}] \tag{1}$$

where R is the wear track depth radius in mm (*2.0 mm in this case*), d is the wear track width in mm, and r is the pin end radius in mm (*1.6 mm in this case*).

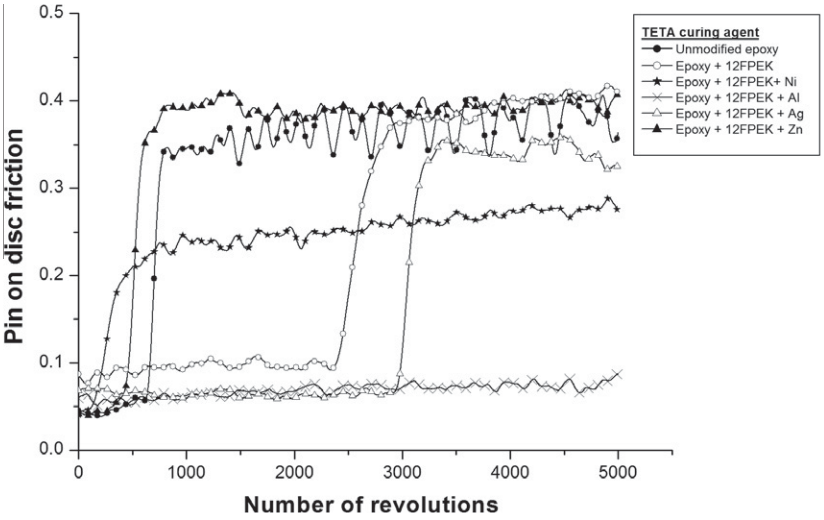


Fig. 2. Pin-on-disc friction vs. number of revolutions of samples cured with TETA.

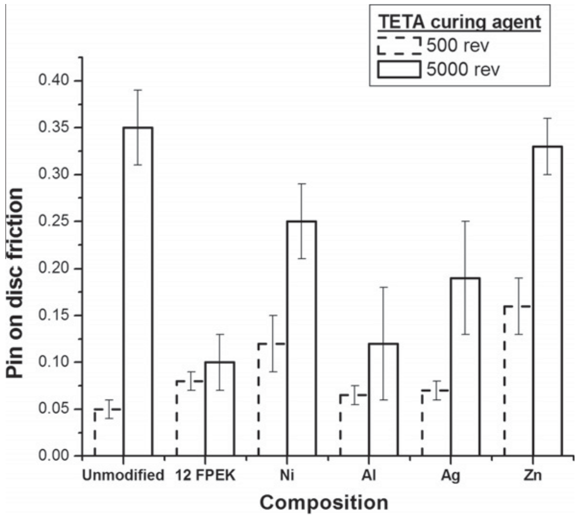


Fig. 3. Pin-on-disc friction after 500 and 5000 revolutions of samples with TETA.

Wear rate (Z) was then calculated using:

$$Z = V_m/WX \tag{2}$$

where W is load in N and X is the sliding distance in m.

2.5. Determination of contact angle and surface energy

In order to determine the surface energies of the samples, first contact angles were determined using sessile drop technique. A Ramé–Hart Instrument Co. goniometer was used for this purpose. A polar liquid (water) and an apolar liquid (diiodomethane) were used as testing liquids [18]. Surface tension values at 20 °C of water and diiodomethane are 72.80 mN/m and 50.80 mN/m respectively. The mean of five contact angles for each sample was calculated.

The Ramé–Hart goniometer which was used for calculating contact angles was used for determining surface energies of the coatings as well. The surface energy values were calculated from the contact angles using the harmonic-mean method [34]:

$$(1+\cos\theta_1)\gamma_1 = 4\{[(\gamma_1^d\gamma_s^d)/(\gamma_1^d+\gamma_s^d)] + [(\gamma_1^p\gamma_s^p)/(\gamma_1^p+\gamma_s^p)]\} \tag{3}$$

$$(1+\cos\theta_2)\gamma_2 = 4\{[(\gamma_2^d\gamma_s^d)/(\gamma_2^d+\gamma_s^d)] + [(\gamma_2^p\gamma_s^p)/(\gamma_2^p+\gamma_s^p)]\} \tag{4}$$

where θ_1 and θ_2 are contact angles made by liquids 1 and 2 on the solid respectively, γ_1 and γ_2 are surface tensions, γ_1^d and γ_2^d are dispersion components of surface tensions of liquids, γ_s^d are dispersion components of surface energy of solid, γ_1^p and γ_2^p are polar components of surface tension while γ_s^p is the polar component of surface energy of solid.

2.6. Surface analysis of coating

Surface composition of the coatings was determined using a Nicolet 6700 Spectrometer with Smart Horizontal ATR micro-sampling accessory from Thermo Electron with 4 cm^{-1} resolution. ATR spectra were collected in the range 800–4000 cm^{-1}. The area of the sample to be measured was selected visually and the spring-loaded ATR element was lowered to make contact with the surface of the sample.

2.7. Metal powder analysis

In order to determine the existence of any secondary phases in the metal powders (as they were used in their pure form) XRD was carried out for all the four metal powders (Ni, Al, Ag, and Zn). This was done using a Rigaku Ultima III High-Resolution XRD. Cu Kα radiation of wavelength 0.154 nm was used. Additional acquisition parameters were: 2θ range = $20^\circ - 90^\circ$ and scan rate = 2° -

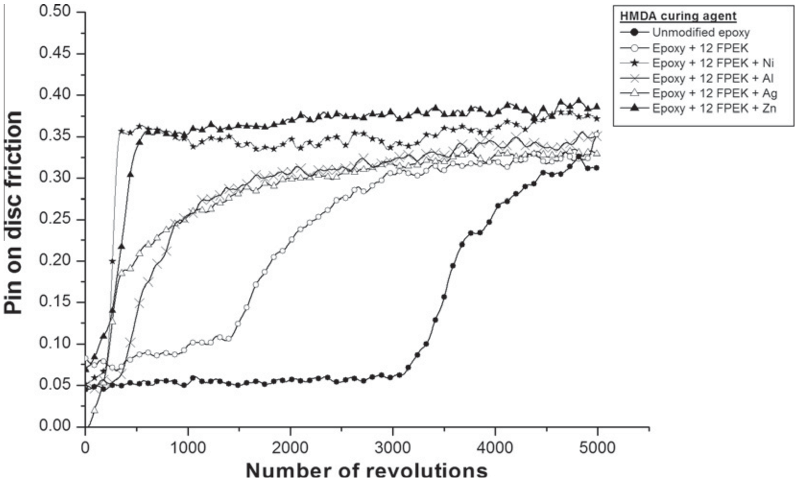


Fig. 4. Pin-on-disc friction vs. number of revolutions of samples cured at 80 °C.

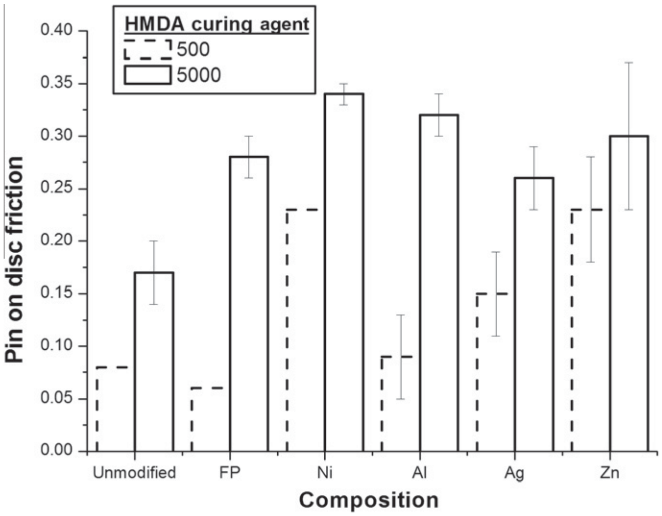


Fig. 5. Pin-on-disc friction after 500 and 5000 revolutions of samples cured at 80 °C.

min^{-1}. Diffraction patterns were referenced against the JCPDS database for sample identification.

3. Results and discussion

3.1. Metal powder characterization

3.1.1. Composition

Since the coatings were prepared in laboratory atmosphere, there was a chance of formation of a new secondary phase within the metal powders like a metal oxide phase which would affect the tribological properties of the final coating. Hence it was pertinent to analyze the composition of the metal powders to determine the existing phases. Diffraction patterns of Ni, Al, and Ag powders did not show traces of any secondary phases but the diffraction pattern of Zn powder shows some peaks related to ZnO phase (Figs. 1a–1d).

3.2. Pin-on-disc friction

Friction increased as the number of revolutions increased from 500 to 5000 irrespective of the curing temperature. To explain this, let us consider the set of samples with TETA curing agent cured at 30 °C. The friction of most of the coatings (except for the epoxy + FPEK + Ni sample) cured at 30 °C was constant up to 500 revolutions. After 500 revolutions the coating on the testing surface got worn out completely and the pin touched the steel substrate; at this point there occurs a sharp increase in friction (Fig. 2) resulting in an increase in friction – continuing up to 5000 revolutions. However, the epoxy + FPEK + Al sample did not get worn even after 5000 revolutions (Fig. 3). Also, friction decreased with the addition of FPEK to the epoxy; during slow curing the FPEK migrates to the free surface since the surface energy of the fluoropolymer is lower than that of pure epoxy [16]. The second set consists

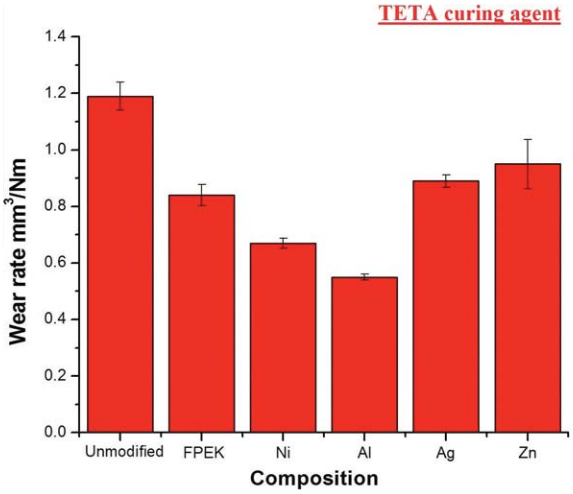


Fig. 6. *Z* of samples with TETA cured at 30 °C.

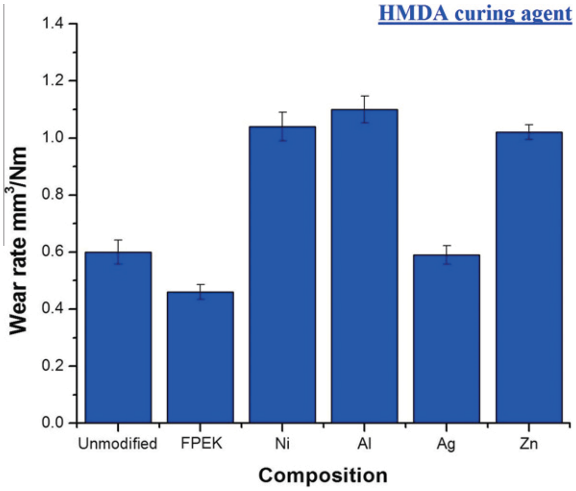


Fig. 7. *Z* of the samples with HMDA cured at 80 °C.

of samples with HMDA post-cured at 80 °C. In this case only the unmodified epoxy sample and the epoxy + FPEK sample got worn out after 500 revolutions. All other samples got worn out before 500 revolutions – the reason for lower friction of the unmodified epoxy sample and the epoxy + FPEK sample compared to other materials (Figs. 4 and 5). This can be explained by the following hypothesis: at high temperatures the crosslinking reaction competes with phase separation and is favored over the latter – resulting in a mixture of epoxy and FPEK phases at the free surface. Samples containing Zn show high friction in both cases – clearly because of the presence of ZnO phase along with Zn phase. Also the morphology of the Zn and ZnO particles is responsible for high friction. Samples containing Al show the least friction when compared to other metal powder containing nanocomposites. This can be explained by spherical morphology of Al particles which results in rolling friction.

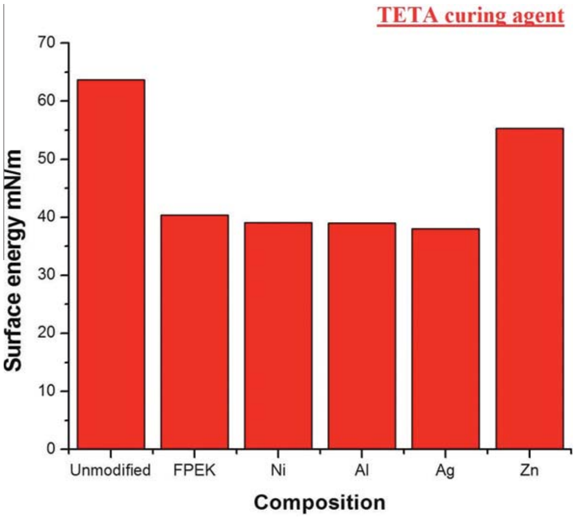


Fig. 8. Surface energies of samples with TETA cured at 30 °C.

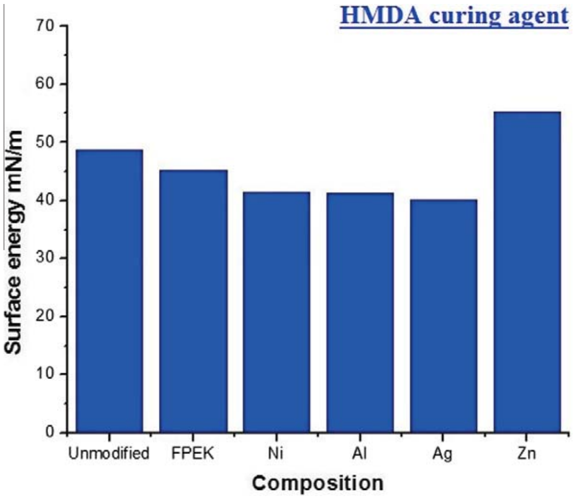


Fig. 9. Surface energies of samples with HMDA cured at 80 °C.

3.3. Wear

Wear results are presented in Figs. 6 and 7, respectively for TETA and HMDA curing agents.

For the samples with TETA we find that *Z* decreases with the addition of FPEK and metal powders. We note that epoxy and FPEK are softer phases while each metallic phase is relatively harder. Samples containing Ni, Ag, and Zn show higher Z values than Al-containing nanohybrids because of the already mentioned spherical morphology of Al. In the materials containing the remaining metal powders, there exists stresses at the interfaces of the particles because of irregular morphologies and hence higher Z values. We recall a discussion by Kopczynska and Ehrenstein of the importance of interfaces for composite properties [35]. We also note that connections between various tribological properties can be pursued [36] while the popular

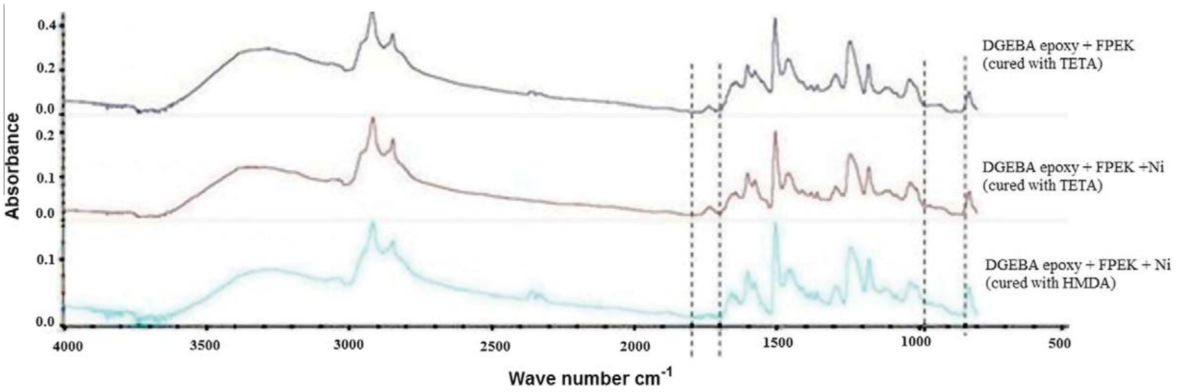


Fig. 10. ATR graph of three samples with epoxy + FPEK + Ni cured at different curing temperatures compared with the epoxy + FPEK sample.

assumption "higher friction means more wear" is by no means generally valid.

In the case of samples with HMDA, since the crosslinking reaction is favored, these systems tend to show higher *Z* values upon addition of the metal powders. The high *Z* of sample containing Zn can be related to the same fact of existence of ZnO phase. Samples containing Ni and Al show high Z values when cured at 80 °C. The test surface of the coating gets worn out as early as ≈250 revolutions in the case of the sample containing Ni and before 1000 revolutions in the case of sample containing Al (Fig. 4).

3.4. Surface energy

Fluorine-containing polymers have low surface free energy, low tendency for water uptake, and high water repellence [15,18,34,37,38]. Therefore, the addition of FPEK to the epoxy should decrease the surface energy significantly. We find that the expected trend is followed; see Figs. 8 and 9. The decrease in surface energy is smaller at the higher curing temperature. The reason is the same as discussed above: as the curing temperature increases, the phase separation process is less pronounced. Therefore, in the case of curing at 30 °C, the FPEK phase is present at the entire free surface. Hence a steep decrease in surface energy. When curing at 80 °C, less fluoropolymer reaches the surface since more of it is trapped by the crosslinking on its way to the surface. In both the cases, the sample containing Zn shows high surface energy consistent with its high friction.

3.5. FTIR-ATR measurements

Three samples: epoxy + FPEK (cured at 30 °C) and epoxy + FPEK + Ni (cured at 30 and 80 °C) were tested for surface composition using FTIR-ATR to analyze the phases present at the surface when cured at different temperatures. The results are presented in Fig. 10. Since characteristic absorbance literature for FPEK was not available, it was pertinent to obtain the ATR for the FPEK sample and then compare the results of the rest of the samples with the FPEK sample to detect what phases were present at the surface of the samples after curing.

There are few new bands which appear when the curing temperature increases. The band at ~1750 cm^{-1} refers to carbonyl group [39] and such a group is present only in FPEK among the materials used. Thus, this band represents the FPEK phase. The band slowly disappears as the temperature increases – what means that the fluoropolymer phase amount is slowly decreasing at the surface as the temperature increases. At the same time, there are new characteristic bands forming between 820 and 980 cm^{-1} as the curing temperature increases; they correspond to the DGEBA epoxy [40]. Apparently, as the temperature increases, there is a mixture of FPEK and epoxy phases that coexist at the free surface. This supports the increasing trend of surface energy and friction with increasing curing temperature. The bands at 3300 and 2900–3050 cm^{-1} correspond to the hydroxyl and C–H groups of DGEBA. There is no change in these peaks in all the four systems corresponding to the presence of DGEBA present near the surface [41].

Acknowledgments

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