Improvement of Interface Interaction and Conductive Anodic Filament Resistance Through Amphiphilic Oligomeric Silane

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ABSTRACT: In this article, an amphiphilic oligomeric silane (OS) was synthesized as a coupling agent to improve the interface bonding between resin matrix and glass fiber. The effect of the OS coupling agent on the interface of glass fiber/epoxy resin was studied by contact angle measurement, gravimetric measurements of water sorption, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and conductive anodic filament (CAF) resistance test. With the addition of the OS to the composites, the contact angle between epoxy resin and glass fiber decrease notably. Normalized water sorption by gravimetric measurements showed that the interfacial debonding time of composites with the OS can be prolonged significantly. CAF tests were also consistent with the water sorption results, which suggest that the gravimetric measurement of water sorption is a cost-effective method to assess the CAF resistance of materials. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 122: 2317–2324, 2011

Key words: interfaces; thermosets; composites; ageing

INTRODUCTION

Interfacial interaction in composites materials is an integrative combination of chemical bonding, hydrogen bonding, molecular interaction, and physical sorption.[1,2] The degradation of fiber/polymer matrix interfacial region, which in fact is an interphase with finite volume, is critical to the performance of the composites materials.

With increasing focus on miniaturized designs and high performance, conductive anodic filaments (CAFs) on printed circuit boards (PCBs) have received much attention in recent years.[3,4] The most common flame retardant (FR-4) type PCBs substrate material consists of an epoxy matrix reinforced with glass fibers. CAF is a result of moisture sorption and consequent electrochemical migration process along separated fiber/epoxy interfaces.[5–7]

Hygrothermal aging plays an important role in the interfacial debonding. The interfacial bond is vulnerable to moisture attack, because water molecules may diffuse to the interface to break the polar attraction and the hydrogen bonding between fibers and matrix. Two possible mechanisms, matrix plasticization and degradation of fiber and matrix interface,[8,9] have been developed to explain the effect of moisture sorption on the composite. In an intensive review of water debonding, Leidheiser and Funke[10] have concluded that a water layer of several monolayers at the interface is responsible for adhesion loss and delamination of polymer/inorganic material composites.

Coupling agents, especially silane coupling agents, have been employed to provide effective chemical bonding between glass fibers and polymeric matrices.[11,12] The effect of various silane coupling agents with different organo-functional groups, like (3-glycidoxypropyl)trimethoxysilane (GPTMS) and 3-aminpropyltriethoxysilane,[13] concentrations,[14] and activation methods[15] on the interlaminar, mechanical, and thermal properties of glass fabric/polymer matrix has been well studied. Yue and Quek[16,17] suggested that the maximum interfacial shear strength is obtained at a critical thickness of the silane layer on the treated glass fibers polymer interface, and then the strength decreases with increase of the thickness of the layer.
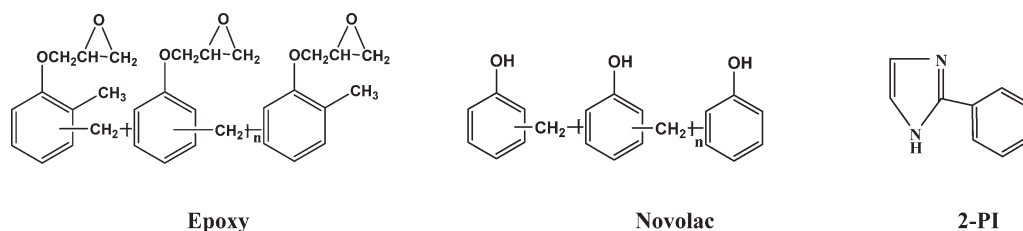
Scheme 1 Chemical structure of the main epoxy resin and hardeners.

Besides directly modification of glass fiber with silane coupling agent, a promising method for interface modification is the use of amphiphilic melt additives. However, this kind of interface improvement merely relies on physical force rather than chemical bonding. With this in mind, in this article, we had combined the advantage of chemical bonding of silane coupling agent and physical interaction of amphiphilic additives to an amphiphilic oligomeric silane (OS) coupling agent.

The amphiphilic OS coupling agent was designed to comprise of a hydrophobic block of alkyl chain with good compatibility to the epoxy matrix, and a hydrophilic block of polyethylene oxide (PEO), which was introduced into the composites to provide both surface wettability and moderate interactions between the glass fiber and the matrix. This OS coupling agent was designed to provide a low contact angle and effective adhesion between the epoxy resin and the glass fiber, which could effectively improve the reliability of PCBs from CAF failure.

EXPERIMENTAL

Materials and specimen preparation

Materials
3-Isocyanatopropyltrimethoxysilane (IPTMS) and GPTMS were supplied by Wacker, dibutyltin dilaurate was supplied by Sinopharm Chemical Reagent, China. Nonyl phenol polyethyleneoxide ether with 4 ethylene oxide was supplied by Dow Chemical and heated in vacuum to remove any water contained before use.

o-Cresol (2-methylphenol) novolac epoxy resin (YDCN-702p from Tohto Kasei, epoxy equivalent: 200–230 g equiv^{-1}), Novolac (H-1 from Sumitomo, hydroxyl equivalent: 107 g equiv^{-1}) and 2-phenylimidazole (2-PI from Shikoku Chemicals Co. Marugame, Japan) used as curing accelerator were depicted in Scheme 1. The E-glass fibers were purchased from Asahi, Japan, and heat cleaned at 450°C for 1.5 h to remove presizing and organic impurities from the glass fiber surface.

Specimen preparation. Stoichiometric amount of nonyl phenol polyethyleneoxide ether and IPTMS were stirred with catalytic amount of dibutyltin dilaurate

at 70°C for 3 h to prepare the OS coupling agent (Scheme 2) before adding into the epoxy.

^{1}H-NMR (400 MHz, CDCl3) δ: 6.70–7.06 ppm (bs, Ar), 4.97(b, ─SiCH$_2$ CH$_2$CH$_2$*NH*─), 3.80 (q, CH$_3$ *CH$_2$*OSi─), 3.50–4.25 (m, ─OCH$_2$CH$_2$─), 3.16 (q, ─SiCH$_2$CH$_2$*CH$_2$*NH─), 2.56 (m, ─Ar*CH$_2$*CH$_2$─), 1.71 (m, ─ArCH$_2$*CH$_2$*─), 1.60 (m, ─SiCH$_2$*CH$_2$*CH$_2$NH─), 1.35–1.30 (m, ─Ar(CH$_2$)$_2$ (*CH$_2$*)$_6$CH$_3$), 1.22 (t, CH$_3$ *CH$_2$*OSi─), 0.96(t, ─(CH$_2$)$_8$ *CH$_3$*), and 0.61 (m, ─*CH$_2$*CH$_2$CH$_2$NH─).

The epoxy resins were cured by the stoichiometric curing agents (epoxy resin: Novalac = 2 : 1 wt %), with 0.5 wt % of 2-PI and 0.5 wt % of silane coupling agents (Scheme 2). For comparison, three kinds of samples were prepared as sample without silane (the control sample) and samples with different silanes herein denoted as *EP-Control*, *EP-GP* (with GPTMS), and *EP-OS* (with the OS of IPTMS and nonyl phenol polyethyleneoxide ether), respectively.

A combination of hand lay-up and compression molding method was used to fabricate laminates nominally 2 mm thick, consisting of 8 plies of glass fabrics. The epoxy resins in acetone were applied onto the glass fabric surface by a hand lay-up technique. After evaporation of the solvent under vacuum, the laminate was compressed thereafter, in a mold (250 mm × 350 mm) at a pressure of 100 bar, and the pressure was applied to the composite at 80°C for 150 min. After fabrication, the glass/epoxy composites were cured at 180°C/2 h, 200°C/1 h and finally cooled slowly down to room temperature for property test.
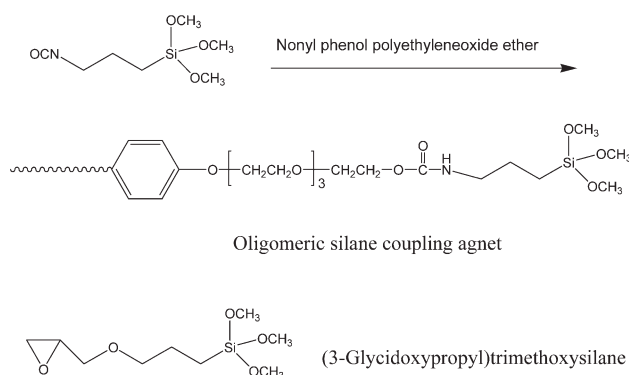
Scheme 2 Chemical structure of the silane coupling agents.

The weight percentage of resin and glass fiber in the prepared composites is 0.683 and 0.317.

Experimental techniques

Contact angle measurements

Dynamic contact angle measurements were performed using a homemade apparatus[18] modified from a drop shape analysis system (DSA100, KRÜSS, Germany). A specific epoxy sample diluted with N,N′-dimethylformamide (DMF in 50 wt %) was sprayed on the glass fiber. A calibrated droplet of water was deposited on the fiber, and the evolution of contact angle with time was recorded using a charge coupled device (CCD) camera with an automatic acquisition of 50 images per second.

Gravimetric measurements of water sorption

Resin matrix (without glass fiber) and composites (with glass fiber) specimens for water sorption experiments were prepared in dimensions of 150 mm × 15 mm × 2 mm, five specimen of the same component were studied for each sample to get the average value of water sorption. Prior to the sorption experiments, all specimens were thoroughly washed and then vacuum dried until a constant dry weight was attained. The medium (distilled water) temperature was kept at 80 ± 0.5°C. The samples were periodically removed from the water, wiped down, and quickly weighed on a Tg332A microbalance (accuracy, 0.01 mg). The water sorption (uptake at time t, M_t) of the sample is achieved as below:

$$M_t = [(W_t - W_0)/W_0] \times 100\% \qquad (1)$$

where W_0 is the weight of the dry specimen and W_t is the weight of the wet specimen at time t.

Diffusion measurements by time-resolved ATR-FTIR

All time-resolved attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) measurements were performed at 20°C using a Nicolet Nexus Smart ARK FTIR spectrometer equipped with a DTGS-KBr detector, solid cell accessories, and a ZnSe internal reflection element (IRE) crystal. The spectra were measured at 4 cm^{-1} resolution and 32 scans, with the wavenumber range being 650–4000 cm^{-1}. The film-covered IRE crystal with a filter paper above the sample film was mounted in an ATR cell, and the spectra of the dry film were collected as background spectra. Afterwards, without moving the sample, distilled water was injected into the filter paper, whereas starting the data acquisition by a macro program. The thickness of the films was about 15 μm.

CAF test

CAF test was performed according to the standard procedure of IPC-TM-650-2.6.25. The samples were placed in an environmental chamber and run up to a steady state condition of 85°C and 85%RH and exposed for 2000 h. During the initial ramp, the temperature was increased ahead of the relative humidity, to prevent any condensation on the samples. Throughout the 2000-h exposure the insulation resistance (IR) was measured.

SEM and EDX observation

A Hitachi (Tokyo, Japan) S-800 scanning electron microscopy (SEM) with a cold field emission source was used to observe CAF morphology on polished sections of the samples. Backscattered electrons were used because compositional contrast is superior to that of secondary electrons. Energy-dispersive X-ray spectroscopy (EDX) was used to analyze the composition of the CAF.

Single fiber pull-out test

Single fiber pull-out tests were performed on an Instron 5576 testing machine. Single glass fibers were end-embedded into the epoxy resin mixture perpendicularly at a preselected embedding length in the range of 50–200 mm. At least 15 samples were cured and studied. Samples were clamped on the tensile stage and the clamps displaced, thus supplying sufficient load to extract the glass fiber from the surrounding matrix.

RESULTS AND DISCUSSION

For surface modification, migration of functionalized additives dispersed in a host polymer to the surface has long been recognized as a potential solution. Recently, silane coupling agents have been successfully added into polymer matrix to improve the interfacial bonding of epoxy[19,20] and other polymers[21] through migration of the silanes. Therefore, in our work, the amphiphilic OS coupling agent was incorporated into the epoxy resin formula with the expectation that it could migrate to the polymer/fiber interface.

Contact angle

As nonyl phenol polyethyleneoxide is always used as a kind of surfactant to improve the surface wettability, here we introduce the PEO block into the silane coupling agent with the expectation of lowing down the contact angle, which would facilitate the wetting process and improve the sorption of the silane coupling agent onto the glass surface. The
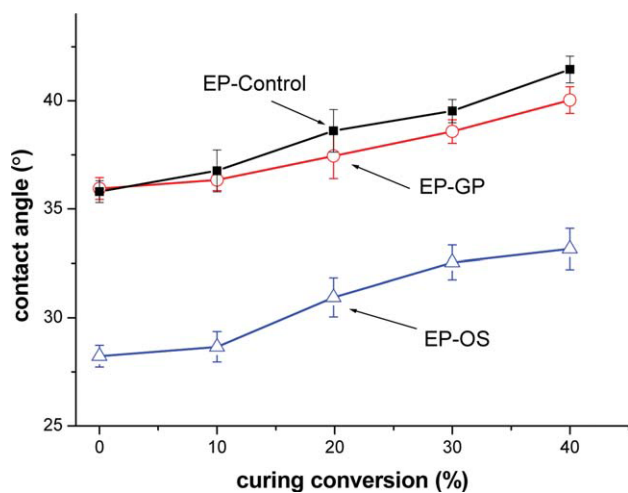
Figure 1 Contact angle on glass fibers as a function of curing conversion in diluted DMF solution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

amphiphilic OS coupling agent was synthesized via a well-known reaction of hydroxyl group with isocyanate. The resultant was characterized by ^{1}H-NMR, where the peaks at 6.70–7.06 ppm correspond to the aromatic hydrogen, 3.50–4.25 to the ether blocks, whereas 3.8 and 1.22 to the siloxane groups.

Figure 1 portrays the relationship between contact angle and the curing conversion of the diluted epoxy solution (in DMF 50 wt %). The contact angles of samples at different curing conversions were measured, which partially reflects the interfacial interaction of fiber and epoxy with increasing viscosity during curing. As one can see, the contact angles of all the epoxy solution on glass fibers are increased slightly with curing. *EP-GP* and *EP-Control* show almost similar wettability, although the addition of GP silane shows a little improvement. Compared with these two samples, the contact angle of *EP-OS* resin solution is effectively lowed down through the whole test, which means that the wettability between the glass fibers and *EP-OS* epoxy resin is much improved. However, it may also induce a negative effect on the hydrophilic PEO block: increasing the polar interactions and/or hydrogen bonding with water molecules. Then another question rose: which is the dominate factor in the interface interaction, surface wettability, or water sorption?

Water sorption

The gravimetric results of water sorption of fully cured samples are shown in Figure 2 and Table I. For comparison, all the samples with and without glass fibers were studied to identify the water absorbed at interface. Apparently, *EP-GP* and *EP-Control* show quite similar water sorption behavior.

However, *EP-OS* samples, no matter resin matrix or composites, show higher water sorption than that of *EP-GP* and *EP-Control*, i.e., the addition of PEO-OS increases water sorption in the epoxy matrix.

According to previous studies and our research work,[22,23] both the free volume and the polarity of
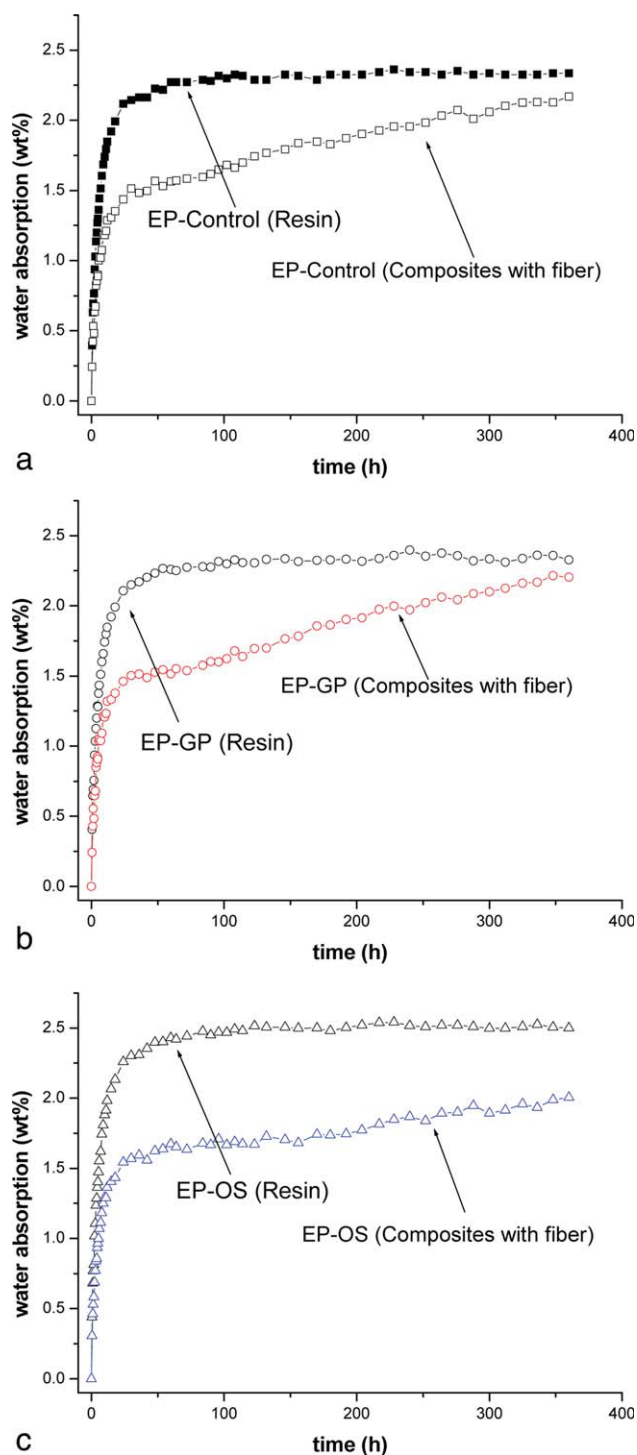

Figure 2 Gravimetric results of water sorption in the samples of resin matrix and composites. (a) *EP-Control*, (b) *EP-GP*, and (c) *EP-OS*. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE I
Calculated Results from Gravimetric Measurements

System	EP-Control	EP-Control composites	EP-GP	EP-GP Composites	EP-OS	EP-OS composites
Water sorption content (%)[a]	2.33	2.17	2.32	2.20	2.50	2.00
Normalized water content (%)	2.33	3.18	2.32	3.23	2.50	2.94
Interfacial water sorption time (h)[b]	–	83	–	90	–	156

[a] The water sorption content is from samples immersed in 80°C water for 360 h.
[b] The time normalized water sorption of resin and composites show clear differences.

the system have effects on the water sorption. In this study, since the only difference between the two silane added systems is the structure of the coupling agent, the free volume of the systems should be about the same. Thus, the change of the polarity of the system could be the key factor that determines the water sorption process.

IR study of the three samples demonstrates the above points. The spectra of water diffusing into EP-OS are shown as an example in Figure 3, in which the IR spectra of absorbed water in the range of 3700–3000 cm^{-1} reveal an increasing intensity of OH variation band (located at 3700–3000 cm^{-1}) as water diffuses into the epoxy networks. Except a slight increase of the band of 1650 cm^{-1}, all the other peaks show little change during the water sorption process. Soles et al.[24–26] suggested that the water molecules would mostly bond to the polar groups in the epoxy resin, and Zhang and Mijovic[27,28] also suggested that above 95% of water molecules would form hydrogen bonds, which meant 95% of water molecules would reside near the polar groups in the epoxy network.

In our systems, with a nonpolar hydrophobic alkyl chain and a polar hydrophilic PEO chain, which has higher polarity than epoxy resin, therefore, the coupling agent improves the polarity of the epoxy matrix slightly, and thus results in a little higher water sorption.

Interfacial debonding

Generally, the water is absorbed into different parts of the composites: the matrix, the interface, and the glass fibers. A mixture rule for water sorption can be given as:

$$M_{wc} = M_{wf}w_f + M_{wm}w_m + M_{wi} \qquad (2)$$

where M_{wc}, M_{wf}, M_{wm}, and M_{wi} are the percent of water absorbed per unit weight of the composites, fiber, matrix, and interface, respectively; and w_f and w_m are the weight fraction of fiber and matrix. As the inorganic glass fiber has minor water sorption, it is obviously that the difference between the water sorption of resin matrix and that of the composite is the interface water sorption.

Therefore, the water sorption at interface can be identified by normalizing the water content of the composite:

$$M_n = M_{wc}/w_m \qquad (3)$$

where M_n is the normalized water sorption of resin matrix, M_{wc} is the water sorption of the composite, and w_m is the weight fraction of resin and coupling agent in the composite.

By comparing the water sorption behavior of resin matrix (without fiber) with that of composites (with fiber) based on eq. (3), one can observe the interface water sorption in this experiment.

Figure 4 and Table I show the normalized water sorption results of EP-Control, EP-GP, and EP-OS. As described above, the difference between water sorption of the matrix with and without glass fiber is the interface water sorption, it can be found that the interface water sorption of the composite EP-Control and EP-GP with coupling agent GPTMS increases with time. On the contrast that of the composite EP-OS with the OS coupling agent is inhibited before 160h and slowly increases as the time prolonged, i.e., the water sorption from interfacial debonding was inhibited due to the existence of OS coupling agent.
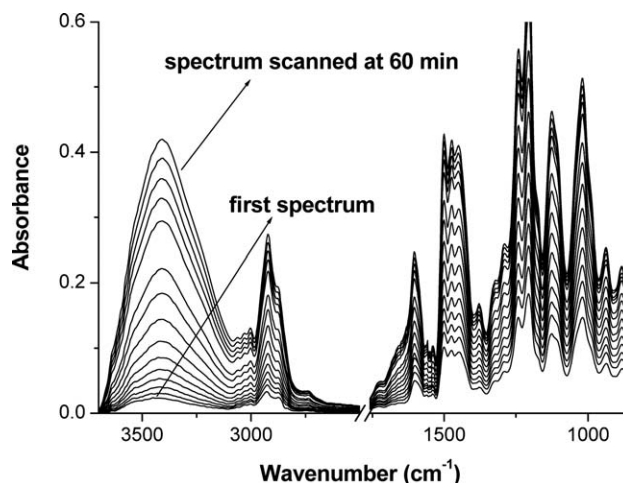

Figure 3 ATR-FTIR spectra of absorbed water in the resin matrix of EP-OS; the time interval for each spectrum is 4 min.
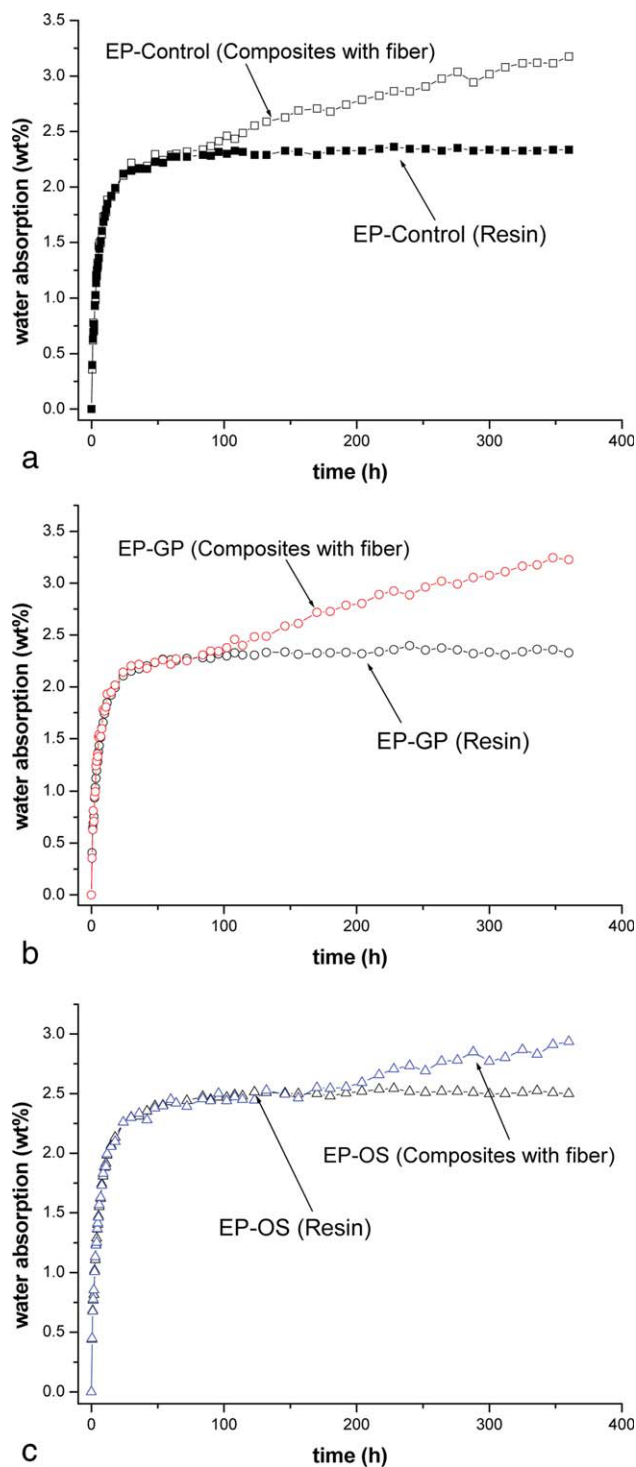
Figure 4 Normalized water sorption in the samples. (a) *EP-Control*, (b) *EP-GP*, and (c) *EP-OS*. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

From these results, it shows that the interfacial adhesion of the composite with the OS coupling agent is well improved, which depresses the interface debonding effectively and results in lower interface water sorption.

CAF test

CAF is the growth or electro-migration of copper in a PCB due to interfacial debonding by hygrothermal aging. CAF test was performed to confirm that the addition of oligomeric coupling agent improves the adhesion between matrix and glass fiber and increases stability of interface.

Figure 5 shows the CAF test result of the systems *EP-Control*, *EP-GP*, and *EP-OS* with the same test condition and parameter of the PCBs. It can be observed that the insulation resistance of both the specimens decreases with test time. However, the insulation resistance of *EP-Control* sample drops below 10^{10} Ω after 1750 h, *EP-GP* is a little better than *EP-Control*, whereas that of *EP-OS* takes place after about 2750 h. This result is quite consistent with the water sorption test and contact angle measurement, i.e., the addition of OS coupling agent can effectively increase the interfacial interaction. Meanwhile as the water sorption is closely linked to the interfacial debonding which is the root cause of CAF, the gravimetric measurements of water sorption could be a cost-effective method to assess the CAF resistance of materials.

Figure 6 shows the SEM and EDX micrographs of at the cross section between two pins where electrical short happened in an epoxy PCB sample prepared according to the standard procedure of IPC-TM-650-2.6.25. In the SEM micrograph, the white round particles are corresponding to the cross section of glass fibers, whereas the gray part to epoxy matrix. The white failure site (indicated by the circle) clearly shows that the filament is confined to a thin region at the separated epoxy/glass fiber interface. Correspondingly, the EDX (copper mapping) in Figure 6(b) shows that the region is where CAF takes place. The lighter regions are
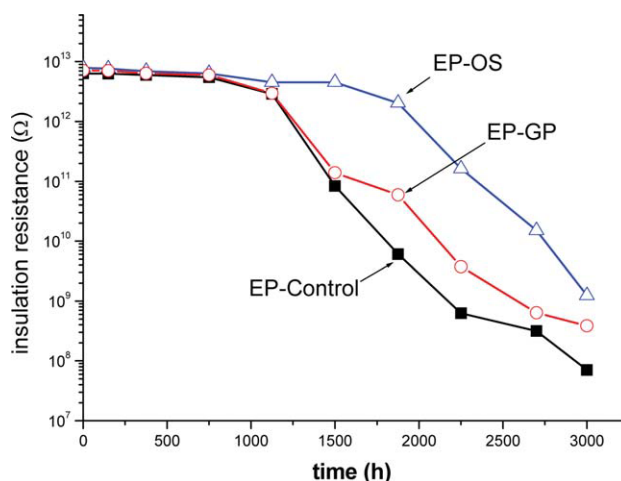

Figure 5 CAF test of PCBs made with *EP-Control*, *EP-GP*, and *EP-OS*. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
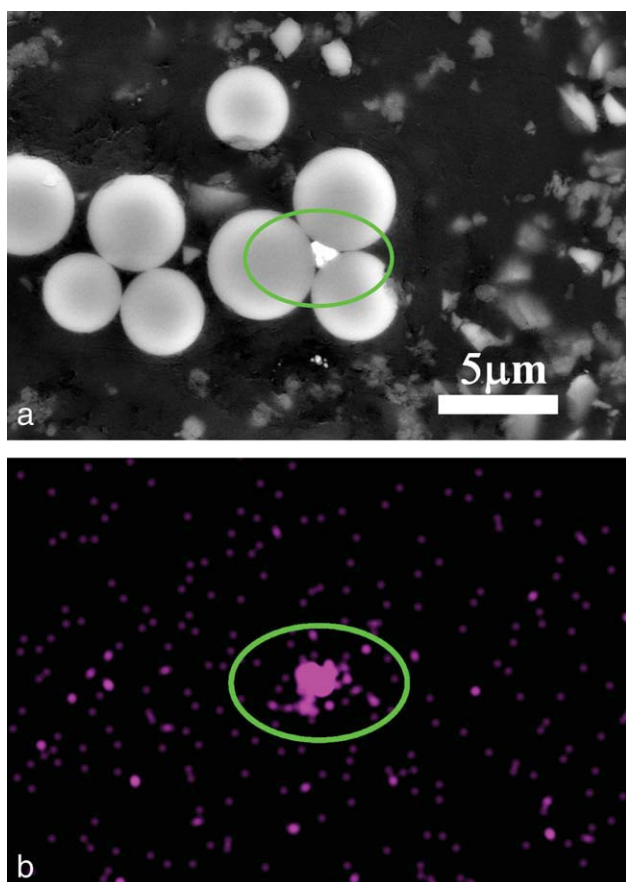
Figure 6 SEM and EDX micrographs for a cross-sectioned site perpendicular to the filament. (a) SEM examination of delamination at the fiber–epoxy interfaces in areas immediately surrounding the failure site. (b) EDX showed copper (red dots) in the shorted location. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

corresponding to higher copper concentrations and the darker regions to lower concentrations of copper. This result confirmed that CAF takes place along the epoxy/glass fiber interface, where copper-containing corrosion by-products emanate from the positively charged anode and progress on the subsurface toward the cathode.

The role of the OS coupling agent

The single fiber pull-out test was used to estimate the quality of interfacial bonding between differently silane added glass fibers/epoxy systems. The apparent interfacial shear strength was tested[29]: τ_{app}, which is determined as $\tau_{app} = F_{max}/(\pi dl_e)$; where F_{max} is the maximum force recorded in the test, d is the fiber diameter, and l_e is the embedded length.

The τ_{app} value of *EP-Control*, *EP-GP*, and *EP-OS* are 41.6, 49.3, and 70.4 MPa (with errors about 15%), respectively. As this parameter strongly depends on the embedded length, and therefore, its mean values can only be used for comparison: it can indicate that *EP-OS* show much better bonding between the components, which includes combined effect of interfacial adhesion and friction, than that of *EP-Control* and *EP-GP*. In other words, the incorporation of PEO block in the OS coupling agent could clearly improve the adhesion between the resin matrix and the glass fibers.

From the above experimental results, it could be deduced that there are two contradictory effects of the amphiphilic OS on the hygrothermal aging of interface interaction, namely, increasing water sorption and improving surface wettability.

On one hand, silane with hydrophilic PEO block would increase the equivalent water sorption of the resin matrix, which might have some negative effect on the interfacial interactions. However, Caputo et al.[30] have demonstrated recently that PEO could complex with copper ions and prevent the formation of CAF.
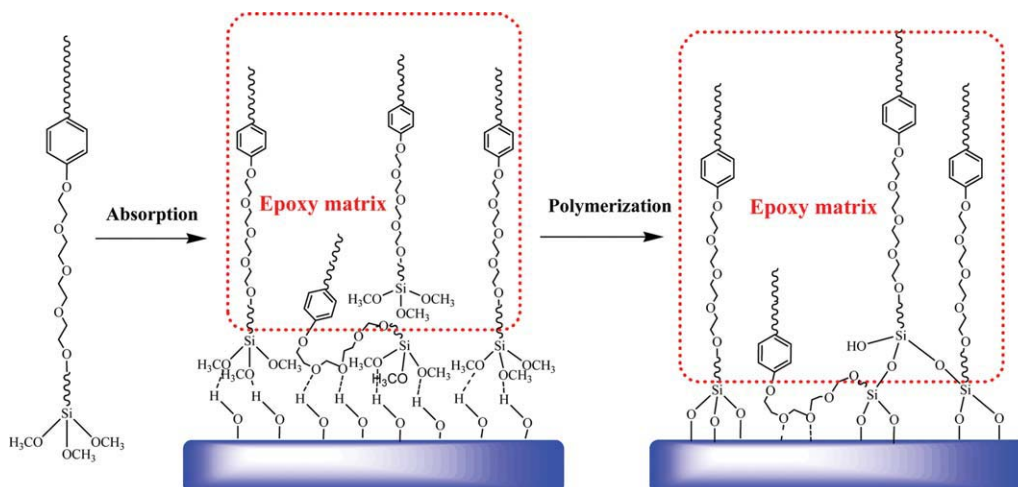
Scheme 3 Schematic representation of the effect of silane coupling agent. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

On the other hand, the amphiphilic OS would low down the contact angle between resin/silane and glass fiber by migration, which would improve the wettability of both the silane and epoxy resin on the glass fiber. The physical sorption of PEO block on glass fiber facilitates the sorption of silane to the interface and thus forms more chemical bonding. As both of the hydrophobic and hydrophilic blocks of the silane have quite good miscibility with epoxy matrix, epoxy resin could wet and spread on the fiber surface filling any micro-voids or air pockets (if unfilled, these would lead to stress concentration and ultimate failure of the composite).[31,32]

Therefore, it can be speculated that adhesion between the glass fiber and matrix is improved by the introduction of the OS coupling agent as illustrated in Scheme 3.

CONCLUSIONS

An amphiphilic OS coupling agent combining the advantage of alkyl chain, hydrophilic block, and siloxane groups could be easily synthesized, and effectively increase the adhesion between glass fiber and resin matrix. The oligomeric coupling agent increases the wettability between glass fiber and epoxy matrix of the composites. CAF test and SEM/EDX shows that the reliability of PCBs can be improved remarkably due to the formation of a stable interface by the oligomeric coupling agent. In addition, the gravimetric measurement of water sorption is a cost-effective method to assess the CAF resistance of materials.

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