# Corrosion Protection of Epoxy-Coated Steel Using Different Silane Coupling Agents

#### JYONGSIK JANG, EUI KYOON KIM

Department of Chemical Technology, Seoul National University, San 56-1, Shinlimdong, Kwanakgu, Seoul, Korea

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**ABSTRACT:** To suppress steel corrosion at elevated temperature and in humid condition, silane coupling agents N- $\beta$ -aminoethyl aminopropyltrimethoxysilane (AAPS),  $\gamma$ -glycidoxypropyltrimethoxysilane (GPS), and bis[3-(trimethoxysilyl)-1-phenylpropyl]tetrasulfide (RC-2) were introduced as primers into an epoxy/steel system. Silane coupling agents and epoxy were coated onto the steel surface using the solution casting method. The polymer degradation and steel corrosion formation after heat and humid treatment were investigated by Fourier transform infrared reflection and absorption spectroscopy (FTIR–RAS) and scanning electron microscopy (SEM). Compared to various silane treated epoxy/steel systems, the AAPS-treated epoxy/steel (AAPS/epoxy = 6 : 4) system suppressed steel corrosion at 400°C for 10 min in air and for 5 days at 60°C in 100% relative humidity. This is due to the formation of Si—O—Si linkage and Fe—O—Si bond on steel surface, which are resistant to water diffusion and thermally stable at elevated temperature. The relationship between chemical bonding at the steel–epoxy interface and corrosion protection on the steel surface was also investigated. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 71: 585–593, 1999

**Key words:** steel corrosion; silane coupling agent; corrosion protection; epoxy resin; siloxane linkage

## INTRODUCTION

When exposed to an ambient environment, most metals corrode through a charge transfer reaction, which causes deterioration at the metal surface.<sup>1</sup> This metallic corrosion causes a tremendous waste of natural resources.<sup>2</sup>

Protective organic coatings are widely used as a primer to protect steel surface from corrosion. However, poor adhesion of coating material to the steel can cause not only delamination of the coating material, but also corrosion of the steel substrate beneath the coating material. The effectiveness of the organic coating depends mainly on the interfacial adhesion strength between metal

Correspondence to: J. Jang.

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and polymer. Thus, proper corrosion inhibitors are needed to extend the service life of steel.

Epoxy is commonly used as the organic coating for steel substrate due to its strong adhesion, excellent chemical resistance, and easy processing.<sup>3</sup> However, epoxy absorbs moisture easily, and the diffusion of absorbed water into the epoxy-steel interface weakens the interfacial adhesion strength between epoxy and steel, particularly in wet conditions.<sup>4,5</sup> To overcome this problem, a silane coupling agent has been used for corrosion protection in an organic coating/steel system.<sup>6-11</sup> Several studies have been undertaken to investigate the effectiveness of silanes for metal corrosion protection with structural adhesives.<sup>6-8</sup>

Jang studied the corrosion protection of steel from an epoxy/DSO (dinorbornene spiro orthocarbonate) system.<sup>12</sup> The epoxy/DSO system pro-

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vided better corrosion protection than epoxy resin due to reduced shrinkage. Consequently, silane coupling agents were introduced as primers, which provide good corrosion protection of steel substrate. Using silane coupling agents as primers caused reduced shrinkage of silane on steel compared to using epoxy. In addition, the Fe-O-Si bond formed during silanization increased the adhesion at steel/silane interfaces. Considered to reaction on epoxy resin, N- $\beta$ -aminoethyl aminopropyltrimethoxysilane (AAPS) and  $\gamma$ -glycidoxypropyltrimethoxysilane (GPS) were selected. Bis[3-(trimethoxysilyl)-1-phenylpropyl]tetrasulfide (RC-2) was chosen because sulfur can make a chemical bond to metal.

This study analyzed the epoxy-coated steel system at elevated temperatures and in humid conditions and also investigated the optimum ratio of silane coupling agents to epoxy for steel corrosion protection. Corrosion behavior and polymer degradation in the epoxy/silane/steel system were analyzed by Fourier transform infrared reflection and absorption spectroscopy (FTIR–RAS) and scanning electron microscopy (SEM).

## **EXPERIMENTAL**

A 1,008 drawing-quality special killed sample was supplied from General Motors Research Laboratories. The chemical composition of the steel sample in weight percent are C, 0.037; Mn, 0.30; P, 0.003; S, 0.002; and Si, 0.005. The steel was aluminum killed and batch annealed to provide a fine grain size. Steel specimens  $(1 \times 5 \text{ cm})$  were mechanically polished with No. 5 chrome oxide. Polished samples were ultrasonically washed in *n*-hexane and ethanol. This was followed by rinsing with 1% HCl/distilled water solution, distilled water, and ethanol, and drying with nitrogen gas.

The silane coupling agents used as a primer were purchased from Dow Corning Co. (Seoul, Korea). They were diluted with acetone. Epoxy resin (EPON 828) was supplied by Kumho Shell Chemical Co. (Seoul, Korea). A solution casting of the epoxy/acetone was applied to coat the steel surface; 14.4 phr diethyltriamine (DETA) was used as a curing agent. The chemical structures of the silane coupling agents, epoxy, and curing agent are listed in Table I.

Film thickness was calculated on the basis of the substrate area, solution concentration, and specific density ( $d_{25} = 1.16$ ) of epoxy resin. Film thickness was fixed at 1  $\mu$ m. The ratios of silane

to epoxy were as follows: epoxy only, silane : epoxy = 2:8, silane : epoxy = 4:6, silane : epoxy = 6:4, silane : epoxy = 8:2, and silane only.

All epoxy-coated samples were cured at 120°C for 40 min. Cured samples were treated at different temperatures for 10 min. In addition, a wet test for cured samples was performed at 60°C in 100% relative humidity as a function of time.

FTIR–RAS was applied to investigate polymer degradation on steel surface and steel corrosion after the elevated temperature and humid condition. The spectrometer was continuously purged with dry nitrogen gas to remove atmospheric water vapor and carbon dioxide. Absorbance spectra were obtained using a Bomem MB-100 spectrometer at a 4 cm<sup>-1</sup> resolution, and 200 scans were collected. A Graseby Specac P/N 19650 monolayer/grazing angle accessory was used. The angle of incidence was 78°, and a freshly cleaned steel coupon was used to obtain the reference spectrum.

SEM was used to observe the corrosion formation of polymer-coated steel plates. The instrument used in this experiment was a Jeol JSM-35, and specimens were coated with a thin layer of gold to eliminate charging effects. The morphologies of polymer-coated steel surfaces after the various test conditions were investigated.

### **RESULTS AND DISCUSSION**

Bare steel was heated at 300, 350, and 400°C for 10 min in air. FTIR-RAS spectra of oxidized steel are shown in Figure 1. The peak at 658 cm<sup>-1</sup> is assigned to  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>, and its intensity increases as heating temperature increases.<sup>13</sup> Because the diffusion rate of oxygen increases with elevating temperature, steel corrosion also increases.

Bare steel was exposed at 60°C for 3 and 5 days in a 100% relative humidity chamber. Figure 2 shows FTIR-RAS spectra of oxidized steel in humid condition. After 3 days, the corrosion was observed. The characteristic peaks at 1020 and 742 cm<sup>-1</sup> are due to  $\gamma$ -FeOOH.<sup>14</sup> The band at 560 cm<sup>-1</sup> is designated as Fe<sub>3</sub>O<sub>4</sub>.<sup>15</sup>

Figure 3 illustrates FTIR-RAS spectra of epoxy-coated steel heated at elevated temperatures. The characteristic peak at 915 cm<sup>-1</sup> of the epoxide group is no longer observed in spectrum A.<sup>16</sup> This indicates that the epoxy resin was completely cured. The band at 1180 cm<sup>-1</sup> is due to the Ar—C—Ar stretching.<sup>16</sup> The band at 1032 cm<sup>-1</sup> is assigned to the aliphatic C—O stretching.<sup>17</sup> The band at 828 cm<sup>-1</sup> corresponds to the out-of-plane



Table I Chemical Structures of Silane Coupling Agents, Epoxy Resin, and Curing Agent

Diethylene triamine (DETA)

#### $H_2NCH_2CH_2NHCH_2CH_2NH_2$

hydrogen wagging of para-disubstituted benzene.<sup>17</sup> In addition, the band at 554 cm<sup>-1</sup> is associated with the out-of-ring bending in para-substituted benzene.<sup>17</sup> At 320°C, the aliphatic C—O peak almost disappear, and the peaks from the benzene ring decrease in intensity due to thermal degradation. At 360°C, epoxy is completely decomposed, and a corrosion peak at 658 cm<sup>-1</sup> is observed. This corrosion peak becomes stronger in intensity at 400°C.

FTIR–RAS spectra of epoxy-coated steel exposed in humid condition are shown in Figure 4. In Figure 4(B), the peaks of  $\gamma$ -FeOOH and Fe<sub>3</sub>O<sub>4</sub> appear at 1,020, 741, and 561 cm<sup>-1</sup>. Due to moisture gain of the epoxy, epoxy-coated steel is sensitive to corrosion in humid conditions. However, the characteristic peaks of epoxy are still observed despite corrosion formation at the epoxy–steel interface. The peak intensity at 1100 cm<sup>-1</sup> from the aliphatic secondary alcohol increased in Figure 4(C) due to increased water absorption.<sup>17</sup>



**Figure 1** FTIR-RAS spectra of steel heated at elevated temperature in air: (A) 300°C; (B) 350°C; (C) 400°C.



**Figure 2** FTIR–RAS spectra of steel exposed at 60°C in 100% relative humidity: (A) 3 days; (B) 5 days.

To protect the steel surface from corrosion, a silane coupling agent was introduced into the epoxy/steel system. Figure 5 shows FTIR–RAS spectra of the epoxy/AAPS/steel (AAPS : epoxy = 6 : 4) system at elevated temperature. Because there is no peak of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> in Figure 5, the epoxy/AAPS/ steel (AAPS : epoxy = 6 : 4) system suppresses the



**Figure 3** FTIR-RAS spectra of epoxy-coated steel heated at elevated temperature in air: (A) the control; (B) 320°C; (C) 360°C; (D) 400°C.



**Figure 4** FTIR-RAS spectra of epoxy-coated steel exposed at 60°C in 100% relative humidity: (A) the control; (B) 3 days; (C) 5 days.

steel corrosion at 400°C. In Figure 5(A), the bands at 1035 and 828 cm<sup>-1</sup> originate from epoxy resin, and the broad band around 1120 cm<sup>-1</sup> is associated with the Si—O—Si linkage and the Si—O—CH<sub>3</sub> group. However, the characteristic peaks of epoxy resin are hardly observed at 1035



**Figure 5** FTIR–RAS spectra of epoxy/AAPS/steel (AAPS : epoxy = 6 : 4) heated at elevated temperature in air: (A) the control; (B) 320°C; (C) 360°C; (D) 400°C.



**Figure 6** FTIR-RAS spectra of epoxy/AAPS/steel (AAPS : epoxy = 6:4) exposed at 60°C in 100% relative humidity: (A) the control; (B) 3 days; (C) 5 days.

and 828  $\text{cm}^{-1}$  in Figure 5(B), due to thermal degradation at 320°C. In addition, the broad band around  $1120 \text{ cm}^{-1}$  is shifted to a higher frequency  $(1151 \text{ cm}^{-1})$ . This peak shift indicates the increase of Si-O-Si linkages through the hydrolysis of Si-O-CH<sub>3</sub> followed by the condensation reaction during the heat treatment.<sup>18</sup> In Figure 5(D), the broad band  $(1172 \text{ cm}^{-1})$  from the infinite siloxane network structure is still observed at 400°C. This siloxane network structure contributes to corrosion protection on steel surfaces at elevated temperatures. Because the amine functional group in AAPS acts as a catalyst of hydrolysis, most of the Si-OCH<sub>3</sub> group in AAPS is easily hydrolyzed, and condenses to form disiloxane bond.<sup>19</sup>

Figure 6 shows FTIR–RAS spectra of an epoxy/ AAPS/steel (AAPS : epoxy = 6 : 4) system in humid conditions. The peaks of  $\gamma$ -FeOOH and Fe<sub>3</sub>O<sub>4</sub> do not show up in Figures 6(B) and 6(C). The Si—OH group in AAPS reacts on Fe—OH on the steel surface, and makes the formation of an Fe—O—Si bond.<sup>20</sup> The Si—O—Si linkage is also formed by the condensation reaction of Si—OH. A formed Si—O—Si bond on the steel surface obstructs water diffusion into the interface between the coating material and steel.

Figure 7 illustrates FTIR-RAS spectra of the epoxy/GPS/steel (GPS : epoxy = 6 : 4) system at elevated temperature. GPC is expected to react

with the curing agent and epoxy. Because epoxy resin is completely decomposed at 360°C, the peak of 830 cm<sup>-1</sup> related to parasubstituted benzene of epoxy disappears in Figure 7(C). The peak of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> appears in Figure 7(D). Judging from the peak intensity around 1121 cm<sup>-1</sup>, the epoxy/ GPS/steel (GPS : epoxy = 6 : 4) system has less Si—O—Si bond (1121 cm<sup>-1</sup>) than the epoxy/ AAPS/steel (AAPS : epoxy = 6 : 4) system. The Si—O—CH<sub>3</sub> group of GPS is slightly hydrolyzed and condenses to form a disiloxane bond during heat treatment, because there is no amine functional group in GPS. The corrosion protection capability of GPS is lower than that of AAPS because of less siloxane bond formation.<sup>21,22</sup>

Figure 8 shows FTIR-RAS spectra of the epoxy/ GPS/steel (GPS : epoxy = 6 : 4) system in humid conditions. The peaks of Fe<sub>3</sub>O<sub>4</sub> and  $\gamma$ -FeOOH are given in Figure 8(B). GPS forms less siloxane bond than AAPS, and hinders water diffusion less than AAPS. Thus, GPS is sensitive to corrosion in humid conditions. The corrosion peaks of Fe<sub>3</sub>O<sub>4</sub> and  $\gamma$ -FeOOH are more intense in Figure 8(C).

Figure 9 shows FTIR–RAS spectra of the epoxy/RC-2/steel (RC-2 : epoxy = 6 : 4) system at elevated temperature. In Figure 9(A), the peak at 780 cm<sup>-1</sup> corresponds to mono-substituted benzene of RC-2.<sup>22</sup> At 320°C, although the peak of benzene from epoxy resin is observed, the peak of benzene from RC-2 almost disappears. This is due



**Figure 7** FTIR-RAS spectra of epoxy/GPS/steel (GPS : epoxy = 6:4) heated at elevated temperature in air: (A) the control; (B)  $320^{\circ}$ C; (C)  $360^{\circ}$ C; (D)  $400^{\circ}$ C.



**Figure 8** FTIR–RAS spectra of epoxy/GPS/steel (GPS : epoxy = 6 : 4) exposed at 60°C in 100% relative humidity: (A) the control; (B) 3 days; (C) 5 days.

to the difference in the position of benzene. The benzene in epoxy is in a polymer backbone, whereas that in RC-2 is in a side chain. The extent of the Si—O—Si formation (1122 cm<sup>-1</sup>) of RC-2 in Figure 9(C) is slightly lower than that of GPS in Figure 7(C). The peak of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> is more intense than that of GPS.



**Figure 9** FTIR–RAS spectra of epoxy/RC-2/steel (RC-2: epoxy = 6:4) heated at elevated temperature in air: (A) the control; (B)  $320^{\circ}$ C; (C)  $360^{\circ}$ C; (D)  $400^{\circ}$ C.



**Figure 10** FTIR-RAS spectra of epoxy/RC-2/steel (RC-2 : epoxy = 6:4) exposed at  $60^{\circ}$ C in 100% relative humidity: (A) the control; (B) 3 days; (C) 5 days.

Figure 10 illustrates FTIR–RAS spectra of the epoxy/RC-2/steel (RC-2 : epoxy = 6 : 4) system in humid conditions. The peak at 1076 cm<sup>-1</sup> is due to Si—O—CH<sub>3</sub> and mono-substituted benzene of RC-2 in Figure 10(A).<sup>17,18,23</sup> During the wet corrosion test, the peak at 1076 cm<sup>-1</sup> decreased and the peak at 1102 cm<sup>-1</sup>, which originates from the Si—O—Si bond and the aliphatic secondary alcohol, increased. Thus, the Si—O—CH<sub>3</sub> group is



**Figure 11** The curve fit spectrum of epoxy/AAPS/ Steel (AAPS : epoxy = 6 : 4).









(C)

**Figure 12** SEMs of bare steel, epoxy/AAPS/steel (AAPS : epoxy = 6 : 4), and epoxy/AAPS/steel (AAPS : epoxy = 6 : 4) after the humidity test: (A) steel surface exposed at 60°C in 100% relative humidity for 5 days; (B) epoxy/AAPS/steel (AAPS : epoxy = 6 : 4) surface exposed at 60°C in 100% relative humidity for 3 days; (C) epoxy/GPS/steel (GPS : epoxy = 6 : 4) surface exposed at 60°C in 100% relative humidity for 3 days. (A), (B), and (C) × 10,000.

converted to a Si—O—Si linkage. From Figure 10(B), the peaks of Fe<sub>3</sub>O<sub>4</sub> and  $\gamma$ -FeOOH are observed, and the extent of corrosion is lower than in Figure 8. Although the extent of disiloxane bond formation of GPS is slightly higher than that of RC-2, RC-2 is more hydrophobic than GPS. This indicates that RC-2 is more resistant to water diffusion than GPS. Thus, the degree of corrosion protection capability of RC-2 is better than that of GPS in humid conditions.

Figure 11 displays FTIR–RAS spectrum of the epoxy/AAPS/steel (AAPS : epoxy = 6 : 4) system. The peaks at 935 and 967 cm<sup>-1</sup> are due to Si—OH. The peak at 1008 cm<sup>-1</sup> is designated to a para-substituted benzene ring. The peak at 1080 cm<sup>-1</sup> is associated with C—NH<sub>2</sub> in AAPS. The band at 995 cm<sup>-1</sup> corresponds to Fe—O—Si, which indicates that the covalent bond is made between steel and AAPS on the steel surface.<sup>24</sup>

Figure 12 shows the SEMs of steel, epoxy/

	Dry Corrosion Test (°C)	Wet Corrosion Test (days)
Epoxy	340	3
(AAPS : epoxy = 2 : 8)	340	$\mathrm{No}^{\mathrm{a}}$
(AAPS : epoxy = 4 : 6)	380	$\mathrm{No}^{\mathrm{a}}$
(AAPS : epoxy = 6 : 4)	400	$\mathrm{No}^{\mathrm{a}}$
(AAPS : epoxy = 8 : 2)	400	$\mathrm{No}^{\mathrm{a}}$
AAPS	380	$\mathrm{No}^{\mathrm{a}}$
(GPS:epoxy = 2:8)	340	3
(GPS : epoxy = 4 : 6)	360	3
(GPS : epoxy = 6 : 4)	360	3
(GPS : epoxy = 8 : 2)	360	3
GPS	340	3
(RC-2: epoxy = 2:8)	340	3
(RC-2:epoxy = 4:6)	360	3
(RC-2:epoxy = 2:8)	360	3
(RC-2:epoxy = 2:8)	360	5
RC-2	340	5

Table IIResults of the Corrosion Test ofEpoxy/Silane/Steel Systems

<sup>a</sup> "No" means that corrosion peaks are not detected.

AAPS/steel (AAPS : epoxy = 6 : 4), and epoxy/ GPS/steel (GPS/epoxy = 6 : 4) surfaces after the wet corrosion test. In Figure 12(A), the white object is the crystal of the corrosion product, which consists of Fe<sub>3</sub>O<sub>4</sub> and  $\gamma$ -FeOOH. The corrosion product is not observed in the epoxy/AAPS/ steel (AAPS : epoxy = 6 : 4) surface, which coincides with the FTIR–RAS spectra. In Figure 12(C), corrosion occurs at the interface between GPS and steel, and the corrosion product delaminates the interface between the coating material and steel. This indicates that the interfacial adhesion strength plays a major role in corrosion protection between steel and the coating material.

Table II summarizes the results of a corrosion test of the epoxy/silane/steel system. The values for the dry corrosion test represent the highest corrosion-resistant temperature at which the peak of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> does not occur. The values of the wet corrosion test denote the day when the peaks of Fe<sub>3</sub>O<sub>4</sub>, and  $\gamma$ -FeOOH first appear. The corrosion-protection temperature of epoxy/AAPS/steel (AAPS : epoxy = 2 : 8) in the dry corrosion test is the same as that of epoxy. This implies that the silane layer is too thin to suppress dry corrosion. This tendency is similar to that of epoxy/GPS/ steel (GPS : epoxy = 2 : 8) and epoxy/RC-2/steel (RC-2 : epoxy = 2 : 8). In a dry corrosion test of the epoxy/AAPS/steel system, the corrosion-protection temperature increases as the AAPS ratio increases. AAPS is hydrolyzed and easily condensed by the catalytic function of the amine functional group within itself. The AAPS layer is effective to make the infinite siloxane network that suppresses steel corrosion at elevated temperatures. However, the AAPS/steel system has a lower value than the epoxy/AAPS/steel (AAPS : epoxy = 8:2) system, which may result from the poorer film property of AAPS as compared to cured epoxy. The defects in AAPS film decrease the corrosion protection capability.

In dry corrosion tests of epoxy/GPS/steel and epoxy/RC-2/steel, corrosion protection temperature also increases with increasing silane content. However, the corrosion protection capability is lower than that of the epoxy/AAPS/steel system, because GPS and RC-2 do not have an amine functional group that can act as a catalyst of hydrolysis and condensation of Si—O—CH<sub>3</sub>. The corrosion protection in the dry corrosion test depends on the amount of Si—O—Si bond.

In a wet corrosion test of the epoxy/AAPS/steel system, the corrosion peak does not appear. The amine functional group in AAPS accelerates hydrolysis and condensation of the Si-O-CH<sub>3</sub> group. AAPS makes an Fe-O-Si bond on the steel surface and resists water diffusion by forming an infinite siloxane network. However, in the other silane systems without an amine functional group, the Si-O-CH<sub>3</sub> group in GPS and RC-2 is less hydrolyzed and condensed than in AAPS. This leads to delamination between epoxy and the steel surface and results in corrosion formation in humid conditions.

## CONCLUSION

Steel corrosion was suppressed in both hot and humid conditions by the addition of a silane coupling agent to epoxy-coated steel as a primer. The epoxy/AAPS/steel system demonstrated the best corrosions protection capability.

AAPS makes the infinite siloxane network, which is thermally stable at elevated temperatures and resistant to water diffusion into the interface between coating material and steel. In the case of AAPS, an Fe—O—Si bond onto the steel surface forms via a chemical reaction between the Si—OH and Fe—OH groups. The epoxy/GPS/steel and epoxy/RC-2/steel systems showed lower corrosion protection capability than the epoxy/AAPS/steel system, due to different functional groups. The epoxy/AAPS/steel (AAPS : epoxy = 6:4) system suppressed steel corrosion at 400°C for 10 min in air, and for 5 days at 60°C in 100% relative humidity.

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