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Preparation and characterization of thermally stable epoxy-titanate coatings

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ABSTRACT

Epoxy-polyamide coatings are used to protect metallic substrates in corrosive atmosphere. Thermal stability of the coating can be improved by the addition of inorganic cross-linking agent. Epoxy resin is incorporated with small percentage of silicone resin and cured with two types of hardeners such as polyamide and butyl titanate. The physical properties, heat resistance properties and electrochemical impedance behaviour of these coatings on steel in 0.5 M NaCl solution have been studied. The result implies that the heat resistant character of the titanate-cured coating is increased from 260 to 370 °C. The impedance study has shown that the coating resistance exerted by both the systems is in the range of $10^5\,\Omega\,\mathrm{cm}^2$ after 6 days of immersion in 0.5 M NaCl. FTIR and Raman spectroscopy analysis confirm the presence of titanate linkage in the cured polymer coating. Thermal stability data indicate that the epoxy silicone resin cured with titanate hardener possesses higher thermal stability than that cured by polyamide hardener.

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1. Introduction

A good organic coating protects steel structures in corrosive as well as industrial environments. Protective coatings with outstanding chemical and physical properties can be obtained from the two pack epoxy coatings [1]. Protective performance of the two pack epoxy coatings depends upon the type of resin, amount of hardener and also the curing temperature. Epoxy resin cured with polyamide hardeners is commonly used to protect new as well as old steel structures, due to their better barrier protection and adhesion over moderately prepared surfaces [2]. However, epoxy based coatings are known to undergo degradation when exposed to UV radiation and high humidity. The amine cured epoxy coatings degrade in the presence of oxygen and UV radiation by photo-oxidation process followed by the loss of gloss and cracking on the coatings [3-5]. Further, the coating has poor thermal as well as flame resistance properties, which limits their application in the field of aerospace and electronic industries. Use of metal complex containing curing agents for epoxy resin had shown better thermal as well as UV radiation resistance [6–8]. To improve the weatherability, heat resistance and UV radiation resistance of epoxy coating, there is a need to modify it with new type of cross-linking hardener [9.10]. Butyl titanate possesses many unique properties, such as low glass transition temperature (Tg), good adhesion and high resistance to thermal oxidation [11,12]. Hence the butyl titanate is chosen as a hardener for the epoxy resin and certain percentage of silicone resin

is also incorporated in the resin part of the composition. Silicone resin formulations are well known to protect the structures from high temperature as well as in humidity environments [13,14]. In this paper the heat resistance performance of the coating of epoxy resin cured with titanate hardener is reported.

2. Experimental

Epoxy resin of Bisphenol-A type with epoxy equivalent 450–500 supplied by Ciba Specialty Chemicals, Mumbai, India was used for this experiment. The epoxy resin was refluxed with 2.5, 5.0, 7.5 and 10.0% of baking type silicone resin (supplied by Wacker Metoark Chemicals Pvt. Ltd., Kolkata, India) for 2 h separately and stored. After 24 h stirring, it was found that the resin samples containing 7.5 and 10% silicone were in turbid condition due to the excess of silicone particles. But the other two resin solutions were in homogeneous form and so were chosen in the blend of silicone resin with epoxy resin. The epoxy resin solution with 5% silicone resin was diluted with xylene to give 50% solution as base part for this study.

The hardener part is polyamide with amine value 210–230 (supplied by Synpol Synthetic Polymers Pvt. Ltd., Ahmedabad, India). Another hardener part is butyl titanate (supplied by M/s Super Urecoat Industries, Ahmedabad, India). These two hardeners were also modified to get 50% solution using xylene as solvent. Mild steel panels of size $10\,\mathrm{cm}\times15\,\mathrm{cm}$, $2\,\mathrm{cm}\times20\,\mathrm{cm}$ and thin sheet of $30\,\mathrm{cm}\times20\,\mathrm{cm}$ size were sand blasted to get Swedish specification SA 2.5 of near white surface profile [15] and used for mechanical properties and corrosion studies. Tin foil of size $10\,\mathrm{cm}\times2\,\mathrm{cm}$ size was degreased with trichloroethylene and used for polymer

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Table 1Mechanical properties and salt spray resistance performance of the polymer coating on mild steel substrate.

S. no.	Properties	Epoxy silicone polyamide (EPS-P)	Epoxy silicone-titanate (EPS-T)
1	Scratch hardness (kg)	1.50	1.50
2	Abrasion resistance (mg. wt. loss/1000 rev/1000 g load)	2.90	2.01
3	Flexibility (Mandrel diameter 3 mm)	Passed	Passed
4	Impact test (kg cm)	54	51
5	Salt spray test (480 h)	1-5 blisters are seen on the surface	1-5 small rust spots are seen on the surface

characterization. The polymer coating of epoxy silicone cured with polyamide hardener is named as EPS-P and that of titanate-cured coating is named as EPS-T. The base part and the hardeners were mixed in the ratio of 70:30 for EPS-P and 80:20 for EPS-T compositions and applied over sand blasted steel panels and degreased tin foils by brush and then dried for 15 days. The coated tin foils were floated on mercury pool and the thin polymer film was taken out and used for FTIR, Raman spectroscopy characterization and thermogravimetric analysis. The mild steel panels coated with epoxy titanate were used for studying mechanical properties, heat resistance characteristics, accelerated test and electrochemical impedance spectroscopic (EIS) measurements. The EIS measurements were carried out with PAR model 6310 system in frequencies from 10 kHz to 100 mHz and the applied signal amplitude was 20 mV. The electrochemical cell used for this study is a three electrode system consists of coated panel as working electrode, a platinum foil as the counter electrode and saturated calomel electrode (SCE) as the reference electrode in 3 wt% sodium chloride solution as electrolyte. The area of the working electrode was 1 cm².

The coating thickness on mild steel panels was measured by using Mikrotester as per the ASTM standard D1004. The panels with coating thickness $30\pm3~\mu m$ were selected for further characterization.

2.1. Mechanical properties

The coated mild steel panels have been subjected to the following mechanical tests:

- (i) Scratch hardness test (ASTM D5178).
- (ii) Taber abrasion test (Federal test method 6192).
- (iii) Flexibility test (British specification no. 3900).
- (iv) Impact resistance test (ASTM G477).
- (v) Salt spray test (ASTM B117).

2.2. Heat resistant property

The temperature resistance property of the coated panels was tested as per the ASTM specification D2485.

2.3. Accelerated salt spray test

A set of coated panels was placed in a salt spray chamber, where 5% sodium chloride solution was atomized by compressed air to create a fog. This test was conducted in accordance with ASTM B117 for 480 h.

2.4. Impedance measurement

The corrosion resistance properties of the EPS-P and EPS-T coated steel surface were evaluated by electrochemical impedance measurements. The impedance measurements were carried out with PAR Electrochemical Impedance Analyzer (model 6310) system over a frequency range of 10 kHz to 100 mHz for applied signal amplitude of 20 mV. The electrochemical cell used for this study

consisted of polymer coated electrode as working electrode, a platinum foil as counter and a saturated calomel electrode (SCE) as reference electrode and 0.5 M NaCl solution as electrolyte. The impedance measurements were carried out periodically for different durations of immersion.

2.5. Fourier transform-infra red spectroscopy (FTIR)

FTIR studies of the polymer films were carried out by using FTIR spectrometer (Naxus-670, UK) to understand the extent to which the butyl titanate curing agent reacts with the epoxy polymer by observing the appearance and disappearance of functional groups present in the polymer film.

2.6. Raman spectroscopy

Chemical composition of coatings can be analyzed by probing molecular vibration using Raman spectroscopy, which is very useful to monitor the amount of double bonds, especially the polarizability of charged during symmetric vibration. The Raman shift of the polymer films was measured by using Reinshow Laser Raman Microscope, U.K. He/Ne laser produced at 632.8 nm.

2.7. Thermogravimetric analysis

This method is used to determine the thermal stability of titanate derivatives and their epoxy coatings. Thermogravimetric (TG), Differential Thermal Analysis (DTA) and Differential Scanning Calorimetric (DSC) measurements were carried out in nitrogen atmosphere with flow rate of 100 ml per minute using SDTQ600, TA instrument US. The DTA and DSC curves were recorded simultaneously.

3. Results and discussion

3.1. Mechanical properties

The mechanical properties of the polymer coating on mild steel substrate were studied on duplicate specimens and the reproducibility of the result was found to be 2.5–4.0%. The mechanical properties of the films on mild steel substrate are given in Table 1. It is found that the EPS-P and EPS-T polymer coatings show more or less similar performance. Slight decrease in value of EPS-T in the impact resistance test indicates that the coating contains excess inorganic particles than the EPS-P coatings, leading to slight decrease in adhesion on the substrate. In general, these two coatings perform well on mild steel substrate.

3.2. Heat resistance test

The high temperature resistance test shows that these coatings at $205\,^{\circ}\text{C}$ for 8 h do not show any color or gloss change on the surface. The test carried out at $260\,^{\circ}\text{C}$ for $16\,\text{h}$ indicates that the EPS-P coated panels have shown slight color change to light brown. This

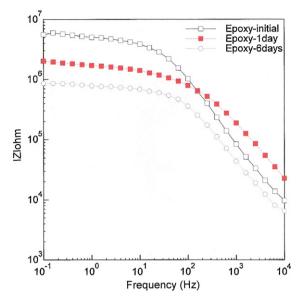


Fig. 1. Bode plot of epoxy silicone-polyamide coated steel substrate in 0.5 M NaCl solution.

indicates that the maximum permissible limit for high temperature withstanding capability of the coating is 260 °C. Micro-cracks were observed on the EPS-P coated panels exposed at 315 °C for 8 h. But the EPS-T coating is found to be unaffected at this temperature. On exposure at 370 °C for 16 h, the EPS-T coating has shown a slight decrease in color, but there is no crack formation on the coating. At a higher temperature of 425 °C for 8 h, the formation of cracks on the coating was observed. Thus these results show that the EPS-T coating is able to withstand up to 370 °C. This heat tolerant behaviour of this coating is due to the presence of titanate linkages (inorganic linkages) in the coating, where as the EPS-P polymer coating contains amide bonding, which is easily breakable at high temperature.

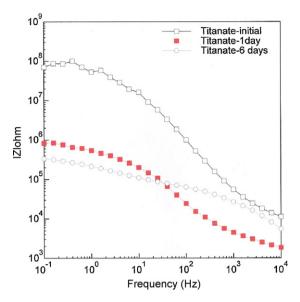


Fig. 2. Bode plot of epoxy silicone-titanate coated steel substrate in $0.5\,\mathrm{M}$ NaCl solution.

3.3. Accelerated salt spray test

Table 1 gives the result obtained from the salt spray test for the polymer film coated on mild steel substrate. From the table it is observed that these two coatings perform well in the accelerated salt spray test for 480 h. Few small blisters are seen on the surface of epoxy-polyamide coatings after 480 h of exposure, where as only a few small rust spots were developed on the surface of epoxy-titanate coated panels for the same duration. It is due to the fact that the epoxy-polyamide coating which has better flexibility property than the epoxy-titanate coatings. Thus the salt fog penetrates into the coatings and lifts it to form blisters on the surface. But the epoxy-titanate coating has moderate flexibility; the salt fog penetrates into it and

Fig. 3. Idealized structure of epoxy-titanate film.

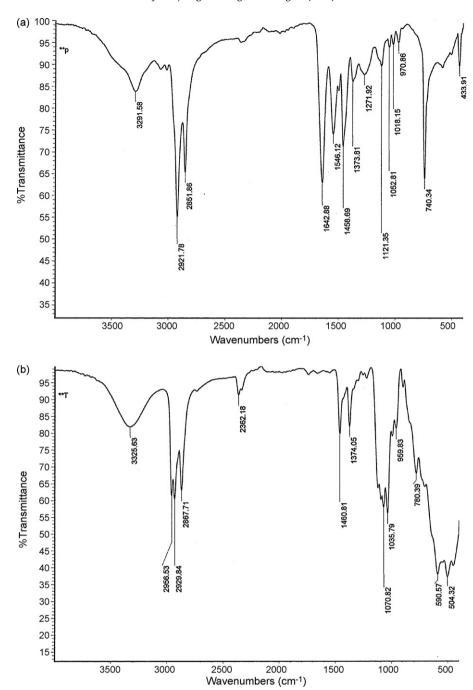


Fig. 4. (a) and (b) FTIR spectrum for polyamide and butyl titanate.

initiates the corrosion process to form rust spots on the surface.

3.4. Electrochemical AC impedance analysis

Figs. 1 and 2 show the Bode impedance plots of EPS-P and EPS-T polymer coated steel in 0.5 M NaCl solution for different immersion time. It is seen from these plots that the resistance offered by these polymer coating has decreased with time. In the initial stage, the EPS-T coating has exerted very high resistance values of $8\times 10^7~\Omega~cm^2$. It is decreased to $9\times 10^5~\Omega~cm^2$ after 24 h in 0.5 M NaCl solution and after 6 days of exposure, the resistance value is of the order of $3\times 10^5~\Omega~cm^2$. This may be due to decrease in rate

of diffusion of water through the polymer after 24 h of immersion and hence the resistance values are not significantly changed. This is mainly due to the presence of titanate linkage in the polymer which has prevented the diffusion of sodium chloride ions. But in the case of EPS-P coating, the resistance value is steadily decreased from 5×10^6 to $9\times 10^5~\Omega$ cm² for 6 days of immersion. This shows that the coating gradually allows the penetration of corrosive ions in to it. This gradual penetration of sodium chloride ions is easier in the presence of amide linkages in the EPS-P coating. The resistance values derived from the Bode plots of duplicate panels are identical with each other with the variation of 5%. Thus the titanate hardener for epoxy resin leads to the formation of strong linkages which prevented the diffusion of corrosive ions significantly.

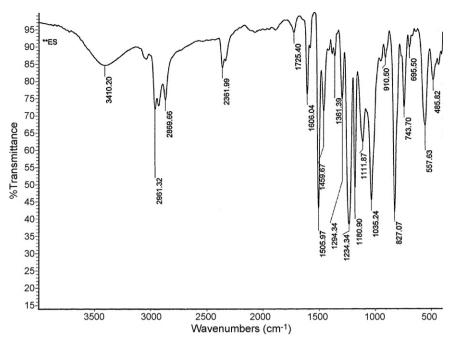


Fig. 5. FTIR spectrum for epoxy silicone resin.

3.5. FTIR spectral analysis

The idealized structure for the titanate-cured epoxy resin is shown in Fig. 3. It is suggested that the titanate group reacts with the epoxy group of the resin to form tetra ether linkages with four molecules of the resin. Additional titanate hardener also reacts with the hydroxyl group of the epoxy resin to form three-dimensional network structures over the steel surface. Fig. 4(a) and (b) depicts the FTIR spectra for polyamide and titanate hardeners, respectively. The polyamide hardener spectrum clearly indicates the presence of amide groups and free amine groups in the molecule. The sharp peaks at 740 and 1642 cm⁻¹ show the presence of excess amide groups in the molecules and also the sharp peaks at 1546 and 1458 cm⁻¹ indicate the presence of free amine groups in the hardener. The broadband in between 3600 and 3200 cm⁻¹ shows the presence of excess free NH₂ group in the molecule. The peaks at 2921 and 2851 cm⁻¹ are assigned to the -CH₂- vibration in the molecules. Similarly in Fig. 4(b), three -CH₂- group stretching frequencies are observed at 2956, 2929 and 2867 cm⁻¹ and the presence of Ti-O group is denoted by the peaks at 2362 and $1035\,\mathrm{cm^{-1}}$. The broadband between 480 and $550\,\mathrm{cm^{-1}}$ shows the presence of titanate ether linkages. Fig. 5 represents the FTIR spectra of epoxy silicone resin. The presence of many functional groups is indicated in this spectrum. The free -OH groups present in the resin is indicated by the appearance of broadband between 3550 and 3200 cm⁻¹ and the presence of aliphatic -CH₂- groups are indicated by the peaks at 2961 and 2869 cm⁻¹. The aromatic -CH=CHare represented by the sharp peaks at 1606 and 1505 cm⁻¹. The presence of phenolic -OH and the epoxy groups are indicated by the peaks at 1234 and 827 cm⁻¹, respectively. The characteristic silicone resin peaks are indicated at 1294, 1180, 1035 and 2361 cm^{-1} corresponding to the groups Si-CH₃, Si-O-Si, and Si-H in the resin. The FTIR Fig. 6(a) and (b) depicts the spectra of cured epoxy siliconepolyamide and epoxy silicone-titanate films, respectively. Since these spectra were taken immediately for films cured for 7 days and so some of the functional groups still appear in the spectra, some of the characteristic groups disappeared or weakened. In Fig. 6(a), the free -OH group band between 3300 and 3200 cm⁻¹

indicates that the OH groups are bonded with hydrogen and so the reaction of this group with other functional groups are minimized. The presence of excess -CH₂- stretches is indicated by strong and sharp peaks at 2922 and 2852 cm⁻¹. The cross-linking of polyamide with the epoxy group is indicated by the appearance of a peak at 1644 cm⁻¹, which is the formation of ester linkage with the polyamide hardener. Further the peak at 2362 cm⁻¹ is weakened due to the reduction of silicone resin concentration. Comparison of the FTIR spectra of titanate-cured epoxy silicone indicates that the titanate and silicone resin peaks appear in more or less same frequency and so after the addition of titanate hardener, the width of the peaks is increased than the polyamide-cured resin. The appearance and broadening of peaks at 2362 and 1104 cm⁻¹ show the formation of titanate linkages with the epoxy resin. Further there is no peak at 1644 cm⁻¹, which denotes that there is no ester linkage in this film, unlike that of epoxy silicone-polyamide coating.

3.6. Raman spectral analysis

Raman scattering provides an experimental tool that is selectively sensitive to changes in bonds and chemical reaction. Fig. 7(a) and (b) depicts the Raman spectra of titanate and titanate-cured epoxy silicone polymer film, respectively. It is observed from the spectra that the titanate group vibration is shifted from 309.42 to 350.89 cm⁻¹. Another shifting also takes place from 170.535 to 171.095 cm⁻¹ [16]. This indicates that a new chemical reaction has taken place between the epoxy group of epoxy resin and the titanate group in butyl titanate leading to new bonding. The Raman shift observed in Fig. 7(a) in the region of 2800–3000 cm⁻¹ is completely disappeared in Fig. 7(b). This is the clear indication of the elimination of butyl group from the molecule. Thus the titanate hardener has formed strong chemical bonding with the epoxy resin. The peaks at higher frequency of 1353 and 1962 cm⁻¹ are due to the -CH=CH- vibration of aromatic groups [17] in the epoxy resin. Thus the results based on the Raman spectra clearly indicate that the titanate group of hardener completely reacts with the epoxy resin to form a new strong bond.

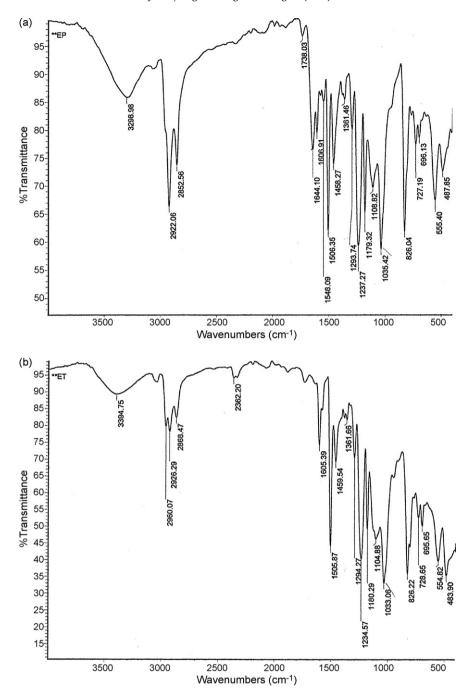


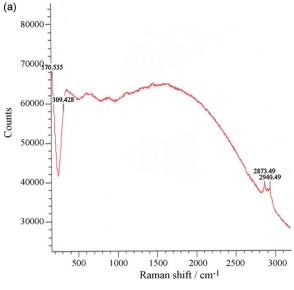
Fig. 6. (a) and (b) FTIR for EPS-P and EPS-T coatings.

3.7. Differential Scanning Calorimetric analysis

Typical DSC spectra of EPS-P and EPS-T polymer films are given in Figs. 8 and 9. The characteristic points of these curves and epoxy silicone resin and the hardener are given in Table 2. It is observed that one endothermic reaction followed by two exothermic transitions are evidenced for resin and hardener and one endothermic followed by three exothermic reactions occurred in the case of cured films. The first transition endothermic reaction Tg occurred at about 50 °C for polyamide hardener, Tg for the cured films is 80 °C for butyl titanate and for epoxy silicone resin it is 90 °C. This is due to the presence of excess inorganic matrix in these formulations. With further heat flow, the polyamide molecule melts and forms further polymerized molecules at 420 °C and liberate 99.17 J/g heat with the elimination of smaller molecules. As the samples are heated

further at about 510 °C, the polymerized polyamide is disintegrated and liberates 1699 J/g heat with the evolution of ammonia and CO₂. In the case of butyl titanate hardener, the two exothermic reactions are found to take place at 250 and 420 °C. In the first reaction stage, the molecular association is taking place with the liberation of butanol and so the heat of liberation is low (90.82 J/g). In the second exothermic reaction, the associated molecules form TiO_2 amorphous powder on the surface and so the heat of reaction is less (109.8 J/g).

The DSC analysis results on the epoxy silicone resin indicate that two exothermic reactions occur in the molecules. The first reaction is at 375.84 $^{\circ}$ C with the liberation of 58.33 J/g, which is due to the reaction of epoxy group in the resin with the methyl group present in the silicone resin to form a strong bond. Further, heat treatment of the resin shows that the liberation of 31.0 J/g of heat corresponds



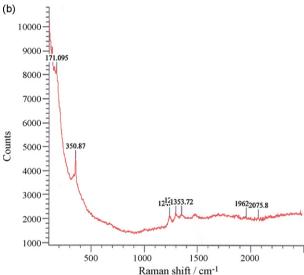


Fig. 7. (a) and (b) Raman spectra for butyl titanate and EPS-T coatings.

to the peak temperature of $551.7\,^{\circ}$ C. This is due to the disintegration of the resin with the deposit of silicate particles and so the heat of reaction is in low order. The DSC analysis of EPS-P and EPS-T indicate the occurrence of three exothermic reactions with more or less similar heat of reaction except in the third exothermic reaction. The peak temperature for reaction of EPS-T is higher than that of EPS-P films. It is evident that the increase in heating rate resulted in the peak position shifts to higher temperature for EPS-T coatings. Thus

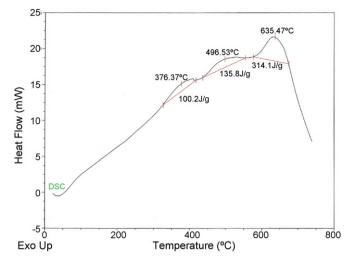


Fig. 8. DSC spectrum of EPS-P coating.

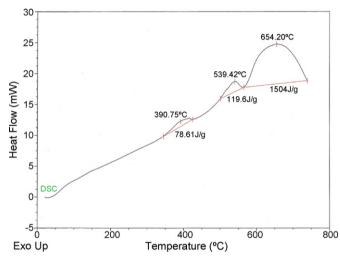


Fig. 9. DSC spectrum of EPS-T coating.

it can be concluded that the reaction of titanate hardener with the epoxy silicone resin increases the thermal stability of the coating.

3.8. Thermogravimetric analysis

The TGA and DTA curves of EPS-P and EPS-T are shown in Figs. 10 and 11. The performance characteristics of the cured films resin and hardeners are given in Table 3. It must be mentioned that the films were flexible and glossy in the initial stages. It can be seen from the thermograms that the primary transition begins at

Table 2Characteristics data obtained from DSC scan of resin, hardener and cured film.

S. no.	Characteristics point	Polyamide	Butyl titanate	Epoxy silicone resin	EPS-P film	EPS-T film
1	Glass transition temperature, Tg (°C)	≈45	≈80	≈90	≈50	≈50
2	 (a) Reaction (i) starting temperature (°C) (b) Reaction peak temperature (°C) (c) Heat of reaction (ΔH) (J/g) 	420 457.97 99.19	250 283.26 90.82	350 375.84 58.33	350 376.37 100.2	360 390.75 78.61
3	 (a) Reaction (ii) starting temperature (°C) (b) Reaction peak temperature (°C) (c) Heat of reaction (ΔH) (J/g) 	510 543.57 1699	420 426.03 109.8	510 551.7 37.31	450 496.53 135.8	520 539.42 119.6
4	(a) Reaction (iii) starting temperature (°C) (b) Reaction peak temperature (°C) (c) Heat of reaction (ΔH) (J/g)	- - -	- - -	- - -	580 635.47 314.1	600 654 1504

Table 3Weight loss data of resin, hardener and cured films from TGA analysis.

S. no.	Weight loss and temperature	Polyamide	Butyl titanate	Epoxy silicone resin	EPS-P film	EPS-T film
1	Temperature TG (°C) Weight loss (%)	45-300 11.98	60–120 10.36	65-340 10.44	45–170 4.61	45–300 9.011
	. ,					
2	Temperature TG (°C) Weight loss (%)	300–500 72	120–300 15.47	340–450 50.01	170–400 27.0	300-450 28.42
3	Temperature TG (°C)	_	300-420	450-640	400-500	450-575
3	Weight loss (%)	-	5.258	11.57	36.78	16.17
4	Temperature TG (°C)	-	580-740	-	500-740	575-740
	Weight loss (%)	-	2.523	-	25.69	27.8
5	Temperature TG (°C)	740	740	640	740	740
	Weight loss (%)	9.959	65.75	27.94	5.941	18.61

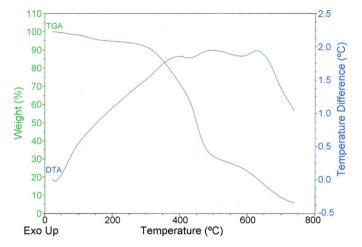


Fig. 10. TG and DTA spectrum of EPS-P coating.

 $170\,^{\circ}\text{C}$ and extends up to $400\,^{\circ}\text{C}$ for EPS-P coating while for EPS-T coating it begins at $300\,^{\circ}\text{C}$ and extends up to $450\,^{\circ}\text{C}$. Above this range there is a sudden decrease in weight for both the systems. Thus the TGA results are nearly consistent with the heat resistance performance of these films, i.e. the EPS-P coating on steel withstands up to $215\,^{\circ}\text{C}$ and the EPS-T coating withstand up to $370\,^{\circ}\text{C}$. Beyond these temperatures, the coatings gradually lose their gloss and begin to develop micro-cracks. This behaviour is also associated with the glass transition temperature of the cured films. However, the weight loss is gradual in the case of EPS-T coatings, while it is rapid in the case of EPS-P cured films. Above $575\,^{\circ}\text{C}$ the weight

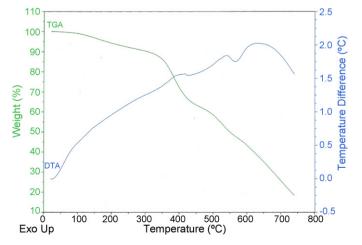


Fig. 11. TG and DTA spectrum of EPS-T coating.

loss is 63% for EPS-P coating and 53% for EPS-T coating. The results indicate that titanate curing shifts the degradation temperature of epoxy silicone film to considerably higher temperatures in addition to reducing the weight loss at elevated temperatures. It is observed from Table 3 that the weight percentage of residue above 740 °C is 65.75 for butyl titanate and 9.96 for polyamide. The performance of the hardeners is also reflected in the cured coatings. These results can be attributed to disintegration of polyamide coating at high temperatures (into ammonia and carbon dioxide) and to the low amount of the residue. In the case of the titanate-cured coatings, the titanate cross-linked films remain on the surface even after the elimination of the organic polymer as $\rm CO_2$ and water.

DTA analysis shows that the EPS-P cured films have three exothermic peaks in the temperature ranges 340-420, 450-580 and 600-700 °C. The first exothermic peak is due to reaction of the epoxy group in the resin with polyamide to form a cross-linked structure. In a subsequent exothermic process, the hydroxyl groups and the silicone resin react with the excess polyamide to form a strong film on the substrate. In the third stage the film disintegrates with release of by-products. Similarly, the EPS-T cured film also has three stages of exothermic reactions. The coating forms an epoxytitanate film in the first stage with the liberation of butanol. In the second stage, the titanate moiety reacts with the silicone resin to form a strong coating on the surface. In the third exothermic reaction the polymer film breaks down. The DTA analysis shows that the curing of the epoxy silicone resin with the titanate hardener considerably shifts the thermal degradation temperature from 340 to 360 °C in the first stage and from 460 to 550 °C in the second stage. This study also reveals that the titanate-cured resin has a higher cross-linking tendency than does the polyamide-cured epoxy silicone polymer.

4. Conclusion

In this study, we have investigated the thermal resistance of the coating formed by epoxy silicone resin cured with butyl titanate hardener. The optimum level of hardener has been identified. Mechanical and heat resistance properties as well as electrochemical behaviour of such films formed on mild steel substrate were studied. Similarly, the thermal resistance of epoxy silicone resin cured with polyamide hardener coating on steel was also studied. Heat resistance study shows that the titanate-cured and polyamide-cured film were stable up to 370 and 260 °C, respectively. Electrochemical impedance studies show that both the coatings exhibited a resistance value of $10^5 \,\Omega\,\text{cm}^2$ after 6 days of immersion in 0.5 M NaCl. The titanate-cured epoxy silicone film has been demonstrated to protect steel structures at high temperatures in corrosive environments. FTIR and Raman spectral analyses confirm the presence of titanate linkages in the cured film, which bestows better heat resistance and weatherability to the polymer film. Thermogravimetric analysis indicates that titanate curing of epoxy silicone resin shifts the degradation temperature from 170–400 to 300–400 $^{\circ}$ C. The TGA data are consistent with those obtained in heat resistance tests. DSC results also support the enhanced thermal stability of the coating formed with the titanate curing agent.

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