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Effect of Addition of Ag Nano Powder on Mechanical Properties of Epoxy/Polyaminoamide Adduct Coatings Filled with Conducting Polymer

Ubair Abdus Samad[a,b] , Rawaiz Khan[a], Mohammad Asif Alam[b], Othman Y. Al-Othman[a,c], Saeed M. Al-Zahrani[b,d]

[a]Department of Chemical Engineering, College of Engineering, King Saud University, P. O. Box 800, Riyadh 11421, Saudi Arabia
[b]Center of excellence for research in engineering materials (CEREM), Advance Manufacturing Institute, King Saud University, P. O. Box 800, Riyadh 11421, Saudi Arabia
[c]Deanship of Graduate Studies, The Saudi Electric University, P. O. Box 93499, Riyadh 11673, Saudi Arabia
[d]SABIC Polymer Research Center (SPRC) and department of chemical engineering, college of engineering, King Saud University, P. O. Box 800, Riyadh 11421, Saudi Arabia

Abstract. In this study the effect of Ag Nano powder on mechanical properties of epoxy coatings filled with optimized ratio of conducting polymers (Polyaniline and Polyppyrole) was evaluated. Bisphenol A diglycidyl ether epoxy resin (DGEBA) along with polyaminoamide adduct (ARADUR 3282-1 BD) is used as curing agent under optimized stoichiometry values. Curing is performed at room temperature with different percentages of Nano filler. Glass and steel panels were used as coating substrate. Bird applicator was used to coat the samples in order to obtain thin film with wet film thickness (WFT) of about 70-90 μm. The samples were kept in dust free environment for about 7 days at room temperature for complete curing. The coated steel panels were used to evaluate the mechanical properties of coating such as hardness, scratch and impact tests whereas coated glass panels were used for measuring pendulum hardness of the coatings. To check the dispersion and morphology of Nano filler in epoxy matrix scanning electron microscopy (SEM) was used in addition Nano indentation was also performed to observe the effect of Nano filler on modulus of elasticity and hardness at Nano scale.

Keywords: Epoxy, Polyaniline, Polyppyrole, Nano indentation, Silver Nanoparticles, Mechanical properties
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INTRODUCTION

Epoxies resins (EP) are widely used in different applications such as; coatings, adhesives, structural reinforcement etc. [1], the major area of application is in coating industry due to its excellent chemical resistance property [2] which make them a material of preference in anticorrosive applications. Besides these advantages EP coatings over a long exposure to environment losses it properties [3]. Many studies have been reported to overcome these limitations. Conducting polymers were found to be attractive as good bearer property material. DeBerry [4] in 1985 reported the exploration of conducting polymer as enhancement of corrosion protection. After that many publications were reported using conducting polymers as corrosion inhibiting materials specially Polyaniline (PANI) [5-8] and Polyppyrole (PPy) [6, 7, 9]. However, these researches were solely concentrated towards anticorrosion properties. The influence of adding conducting polymer on final properties of coatings has been reported by Elaine et al. [10] regarding retardation in mechanical properties by incorporation of PANI and PPy. Some other works have also been reported with modification using nanoparticles (NP) such as ZrO_2 and ZnO to improve final coating properties [11-12].

This study aims to evaluate mechanical properties of EP-PANI (0.78 wt. % PANI) coatings and EP-PPy (0.95 wt. % PPy) coatings with and without silver nanoparticles in epoxy matrix to perceive the effect of nanoparticles on coating's final properties.

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EXPERIMENT

Materials

Dighycidyl Ether Bisphenol A type epoxy resin was purchased from Hexion Chemicals, polyaminoamide adduct (ARADUR 3282-1 BD) curing agent was purchased from Huntsman Advanced Materials, PANI (catalog number: 561126), PPy (catalog number: 530573) and silver nanoparticles (Ag-NP) (catalog number: 576832) were purchased from Sigma-Aldrich and acetone (Merck, 99%) was purchased from local Saudi Arabian market.

Preparation of Coating

Conducting polymer (CP) (PANI and PPy) was added directly in epoxy matrix (EP) in presence of acetone (for ease of mixing). Acetone does not affect any chemical changes in epoxy network during mixing and sonication [13]. EP and CP were mixed using Disper Master S2 (Sheen Instruments, Ltd. UK) under constant stirring at 3000 rpm for 1hr. To dissipate heat generated while mixing at high rpm cooling bath was used. On the other hand, Ag-NP were mixed in acetone with silane as a coupling agent for better adhesion. The prepared Ag-NP solution was then added to EP/PANI and EP/PPy mixture and mixed for 1hr at constant 3000 rpm using mechanical stirrer. After mixing hardener was added in solution at 500 rpm and left for stabilization up to 10-15 minutes. The details of coatings composition is described in table 1.

TABLE 1. Various EP formulation with PANI (0.78 wt. %) and PPy (0.95 wt. %) and their modification with Ag-NP (0.5 wt. % and 1.0 wt. %)

Sample code	EP (wt. %)	CP	Ag (wt. %)	Curing Agent (wt. %)
ECP1	82.68	PANI	0	16.53
ECP2	82.26	PANI	0.5	16.45
ECP3	81.85	PANI	1.0	16.37
ECP4	82.54	PPy	0	16.50
ECP5	82.12	PPy	0.5	16.42
ECP6	81.70	PPY	1.0	16.34

Coating on Samples

Samples (such as glass and steel panels) were degreased using acetone. A four sided Bird applicator (sheen instruments, Ltd. Uk) with micrometer (μm) gaps sizes (30/60/90/120) was used to coat the samples, gap size of 60 and 90μm was used for glass and steel, respectively. The coated samples were left for 7 days for complete curing at room temperature. After curing dispersion analysis was performed using SEM (JEOL JSM7600F). The samples were mounted on aluminum stubs using carbon tape and coated with thin gold layer to avoid charging of the samples.

Mechanical Properties

Pendulum hardness tester (BYK-Gardner Germany) was used for determining konig hardness while nano indenter (Micrometerials. Co. UK) was used to measure the nano-hardness and elastic modulus of the EP coatings. For nano properties measurement using nano indenter load control program was used with maximum load of 250 mN using Berkovich type indenter, at least 25 indentations were performed on sample at different positions for confirmation of results. Automatic scratch tester (Sheen REF. 705) was used for determining scratch resistance of EP coatings by applying different loads (0.5 – 10 kg) on the coated panel with the help of scratch probe. Sample is adjusted on machine holder with some load applied on probe using standard weights, turning on the machine automatically drives scratch probe on the coating surface while applying scratch load simultaneously. The test was repeated with increasing load until scratch probe damages the coating surface. The weight at which coating is damaged is recorded as failure load. Impact test was made using Gardner-SPI Modified Impact Tester (ASTM D2794) in which a standard weight of 8 lb. was dropped from a certain height that damages the coating along with the substrate.

RESULTS AND DISCUSSION

Morphological study

Figure 1 shows the dispersion analysis of Ag-NP in EP/PANI and EP/PPy coating. Fig. 1 (a) and (a'-a'') represents ECP1, ECP2 and ECP3 coatings, respectively. Whereas Fig. 1 (b) and (b'-b'') represents ECP4, ECP5 and ECP6 coatings, respectively. Figure 1(a) showed smooth coated surface and good dispersion of PANI in epoxy matrix while in figure (a') showed low scale agglomeration of Ag-NP with smooth surface, in figure (a'') rough surface with small cracks were observed with higher agglomeration. Fig.1 (b-b'') showed smooth coated surface with good and homogenous dispersion of Ag-NP along with PPy in resin matrix with no signs of surface voids.

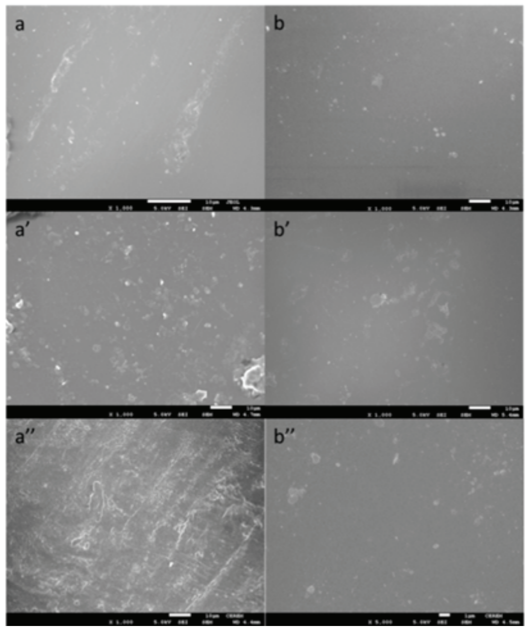

FIGURE 1. Surface area SEM morphology (a-a'') for ECP1- ECP3 and (b-b'') for ECP4- ECP6

Mechanical Properties

Table 2 shows Konig pendulum hardness on glass coated panels, while scratch and impact strength results on steel coated panels with thickness of 70-90 μm. The results indicated that Ag-NP embedded coatings (such as ECP2 and ECP3) have better hardness as compared with ECP1. Whereas, addition of Ag nano particles reduced the scratch and impact strength. The decrease in scratch resistance and impact strength can be attributed due to the agglomeration of Ag-NP and roughness on the surface of Ag modified coated sample [14], this results also confirmed from Fig. 1(a'-a''). This might due to the agglomeration of Ag-NP discussed earlier in figure 1(a'-a'').

TABLE 2. Mechanical properties of coated panels with various coating formulations

Formulation	DFT (μm)	HARDNESS (Oscillations)	SCRATCH (Kg)	IMPACT (lb/in^2)
		EP/PANI		
ECP1	70 – 90	58	8	112
ECP2	70 – 90	107	7	94
ECP3	70 – 90	117	6	64
		EP/PPy		
ECP4	70 – 90	140	4.5	48
ECP5	70 – 90	130	7	80
ECP6	70 – 90	120	8	136

In case of EP/PPy samples the results indicated that Ag-NP embedded coatings (such as ECP5 and ECP6) have slightly lower hardness as compared with ECP4. Whereas, better results were observed for scratch resistance and impact strength. This improvement in properties are linked to homogenous dispersion of Ag-NP in bulk (as discussed earlier in Fig. 1(b'-b'')).

Table 3 and Fig. 2 show the nano-hardness and elastic modulus obtained from Nano indentation. Result indicated that ECP2 and EPC3 coating have improved hardness while elastic modulus is slightly retarded with increasing amount of Ag-NP, while in case of ECP5 and ECP6 coating, approximately similar hardness was observed, however there is an increase in elastic modulus that could correlates with the improvement of scratch and impact properties.

TABLE 3. Hardness and Reduced modulus of various coating formulations modified by Ag nano particles

Sample code	Hardness (Gpa)	Reduced Modulus (Gpa)
ECP1	0.17455	4.1278
ECP2	0.17912	3.9677
ECP3	0.18906	3.6012
ECP4	0.17004	4.0348
ECP5	0.16601	4.2826
ECP6	0.15996	4.3372

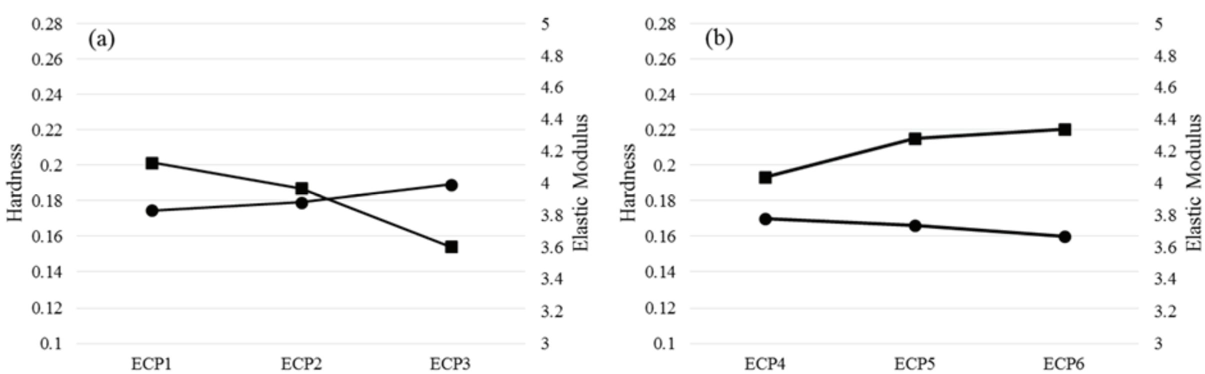

FIGURE 2. Effect of Ag-NP on (•) hardness and (▪) Elastic Modulus on epoxy coatings such as (a) EP/PANI and (b) EP/PPy

CONCLUSION

Different percentages of Ag Nano powder were added to an optimized EP/PANI and EP/PPy coating formulations. Various mechanical properties including Konig hardness, scratch resistance, impact resistance, Nano hardness (indentation) and Elastic modulus were studied. Results indicated that as the Nano powder loading in EP/PANI formulation increased, the resistance to scratch and impact decreased while Konig hardness and nano hardness both increased with increasing the percentage of Ag in epoxy matrix. On the other hand modulus of elasticity decreases for EP/PANI modified coatings which concludes that addition of Ag in EP/PANI induce brittleness in coatings. Whereas, in case of EP/PPy coating formulations addition of Ag leads to improvement of resistance to scratch, impact strength and modulus of elasticity while small retardation was witnessed in hardness values. However, the values are still in acceptable rages. SEM results illustrated that the dispersion of the Nano powder in the polymer matrix is a great challenge when going for higher loading. The dispersion of the Nano powder has great effect on the mechanical properties of the coatings.

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