

Characterization of steel lined with multilayers of micro/nano-polymeric composites

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Article

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Abstract

A more cost-effective method to prevent steel structures from degradation is coating. This study investigated a comparison between different configurations of three multilayers polymeric coating on both sides of steel. Epoxy filled with 1 wt%, 2wt%, and 3wt% micron or nano-sized alumina (Al_2O_3) particles represents the coating layers to steel on both sides. Mechanical and barrier resistance properties were conducted on the micro/nanocomposite coated specimens and compared with pure epoxy coated specimens. Barrier resistance was performed by immersing the lined steel specimens in salt solution and in citric acid medium. Adding alumina (Al_2O_3) particles in micron and nano size to epoxy coatings improves the barrier resistance, the tensile and hardness under dry and wet conditions as compared to pure epoxy coating. The maximum improvement of the tensile strength and barrier resistance was attained with the addition of 1wt% Al_2O_3 nanoparticles. Further increases in Al_2O_3 micro/nanoparticles cause deterioration in tensile strength and barrier resistance. Good properties were observed with stepwise graded micro/nanocomposite coatings. However, the maximum improvement of hardness under dry and wet conditions improved with the further addition of 3wt% Al_2O_3 nanoparticles to epoxy coatings.

1. Introduction

The corrosion of metal is considered as one of the vital issues for steel structures when these structures are subjected to corrosion [1]. Steel has high mechanical strength with low cost fabrication. Consequently, it is utilized in drilling equipment, ship building, and pipelines. In marines, corrosion results in 30% of the total failure thus needed to be repaired or replaced partially. In a marine environment, the corrosion of steel is influenced by salinity and alkalinity [2]. Subsequently, coating was performed on steel faces to avoid the corrosion of new or existing steel construction. The corrosion of steel attracted many research interests as it is costly particularly in oilfield and marine environments [3]. Recently, polymer composite liners to steel was used to decrease diffusion of oxygen and moisture. The protective organic coating as epoxy coating to metal is characterized by their excellent weather ability [4]. Protected epoxy coating has attracted great attention in wet environment due to its very good toughness, durability and adhesion to metal substrates [1]. However, the highly cross-linking density and the barrier behavior of the epoxy coating can be undesirably affected when exposed to corrosion. The polymer coating weakening results in the creation of holes and defects in the epoxy coating surface. During exposing to corrosive media, holes and defects become larger in width and depth. Holes are considered as conductive paths as the electrolyte diffuse in the polymeric coating [5]. Moreover, the protective coating fails by the cause of delamination which is the separation at the polymeric coating/metal interface [6]. The deterioration of polymeric coating decreases the barrier properties thus the mechanical properties of the polymeric coating [5]. Therefore, it is essential to enhance the properties of epoxy resin by replacing epoxy with epoxy composite coatings to achieve the requirements of real applications [4].

Embedded of inorganic fillers to epoxy coating is one of the methods to enhance anti-corrosion characterization of organic polymeric coatings. Adding smaller filler particles in micron or nano size may improve barrier properties of the introduced polymeric coating. Size, morphology, shape, and the weight percentage of the fillers greatly affect the intrinsic characteristics of composite [2]. Nanoparticles are considered as a good water barrier and thus effectively obstructs water absorption improving the service life of metals [2]. Different nanomaterials are involved at various levels in the food industry having both positive and negative effects towards human health. Alumina can also be present due to contamination or migration from other food contact materials such as processing machinery, utensils, and devices [7]. The coatings containing Al_2O_3 particles showed enhancement in scratch and abrasive resistance compared with that of polymer coating. This enhancement in scratch and abrasive resistance is attributed to the dispersion hardening of Al_2O_3 nanoparticles in polymer coatings [8]. Enhancement in environmental impact can be attained utilizing nanosized particulates in polymeric coating and eliminating the requirement of toxic solvents [9]. Nanoparticles embedded in polymeric coatings are well known for their outstanding physical, mechanical and thermal properties [10], [11].

Ramezanzadeh and Attar [5] investigated the corrosion resistance of the epoxy coating containing micron and nano sized ZnO fillers. The specimens were submerged in 3.5 wt% NaCl solution. The corrosion resistance of the coupons was significantly decreased after immersion for 15 days. The corrosion resistance of the epoxy coating was enhanced as reinforced with nano sized ZnO fillers. The results showed that the lowest reduction in cross-linking density and reducing hardness of the polymeric coating submerged in 3.5 wt% NaCl solution were attained as the epoxy coating was reinforced with the 3.5 wt% nano ZnO particles. Moreover, the adhesion was also increased at 3.5 wt%. Furthermore, Anaki and Xavier [1] studied the dispersability of reinforcing epoxy coating on mild steel with 2wt% of nano Al_2O_3 . The resultant specimens has been submerged in 3.5% NaCl solution. The improved anticorrosion performance was conducted by the modified nanocomposite coating as compared to epoxy coating. The reinforced epoxy coating resulted in good adhesive strength, increasing in hardness, tensile strength, and better corrosion resistance than epoxy coating. In addition, Golru et al. [12] prepared epoxy/polyamide reinforced with 1, 2.5 and 3.5 wt% nano-alumina filler coted AA1050 substrate. The results showed that the nanofillers dispersed uniformly in the polymeric coating even when loading at high percentages. The polymeric coating corrosion resistance was more improved by increasing the weight percentage nanofillers.

In recent times, multilayered nanocomposites have gained a great attention due to their required characteristics as microwave absorbing, mechanical properties, permittivity constructed on the interfaces between adjacent layers and the synergistic impacts of fillers. Nevertheless, the application of multilayered of micro/nanocomposite coatings has not been reported yet [4]. Al_2O_3 filler in micron size is commercially available and has a lower cost than Al_2O_3 in nano-size. So, the objective of the study is to develop multilayers of epoxy liners to steel filled with micro and nano Al_2O_3 particles with different percentage and differentiate between them. Three percentages of alumina micro and nanoparticles (1 wt%, 2wt%, and 3wt%) were introduced to epoxy with different configurations. The specimens were

immersed in salt solution and in citric acid media. Barrier resistance and mechanical properties were investigated under dry and wet conditions.

2. Experimental Work

2.1 Materials

Mild steel was used as a metal substrate supplied from Al Ezz-Dekheila Steel Company Alexandria. The steel sheets were cut to the required dimensions of the specimens by a laser machine. The specimens were polished in order to roughen the surface of the steel substrate. After polishing, the top and bottom side of the coupons surface was cleaned with acetone before coating. Chemicals including sodium hydroxide, citric acid and acetone were supplied by El Nasr Pharmaceutical Chemicals, Egypt. The coating is Epoxy resin (Kemapoxy RGL150) which is supplied by CNB Company, Egypt. The reinforcements are Al_2O_3 fillers in micron and nano size with purity about 99%. The size of microparticles and nanoparticles are 90 μm and 70 nm, respectively.

2.2 Preparation of micro/nanocomposite protective films

Epoxy protective films was made by adding hardener carefully to the epoxy and blended thoroughly with a ratio of 1:2 by mass of epoxy resin. The micro/nanocomposite protective films were performed as micro, or nanoparticles were added to the epoxy resin by sonication process. The sonication was performed with Hielscher ultrasonic processor UP 200 S. The sonication was conducted at 0.5 cycles per second on/off with amplitude 40% for 2 hours as recommended by [13], [14]. For epoxy resin protection from degradation, the mixture were cooled by placing it on an ice water bath during sonication [15]. Afterwards, the blend and the hardener were mixed with the recommended ratio in a temperature of 25°C. The protective layer was prepared on steel by a metallic roller to remove excess resin, to reduce void content and any entrapped air bubbles. Painting on one side of steel specimen is left for 24 hours to cure. Subsequently, the second layer on the same side was constructed and left for a day to cure. and the same with the third layer. The same technique was done for the other three layers on the bottom face of the coupons. The final stepwise graded and non-graded micro/nanocomposites coatings on steel substrate were constructed as illustrated in Fig. 1.

2.3 Experimental Procedure

2.3.1 Tensile test

The tensile behavior of the steel coated with micro/nanocomposites coupons were tested according to ASTM D3039. The tensile test was achieved with a computerized universal testing machine (Jinan Test Machine WDW 100 kN). The cross-head speed were set at 2mm/min and the stress–strain curve was recorded by a computer data acquisition system. All tests were performed at ambient temperature.

2.3.2 Hardness

The hardness was determined via the PCE-1000N Hardness instrument measured at ten different places of the micro/nanocomposite coated steel and the average value was taken.

2.3.3 Barrier properties

Some test coupons were immersed in salt solution and in citric acid solution to estimate the corrosion media of the steel lined with micro/nanocomposite. Salt solution was performed as 3.5% NaCl dissolved in water. Citric acid solution with Concentration of 2 N was prepared by double distilled water. Uptake tests were performed according to ASTM D5229 / D5229M - 14. The coated coupons were periodically withdrawn from the solutions, wiped dry, and weighed using an analytical balance of accuracy up to 10^{-4} g to monitor the weight change during the absorption process. The solution content $M(t)$ absorbed by micro/nanocomposite protective coating was then calculated as the mass gain percentage referring to its initial weight (w_0) as follows [16]:

$$M(t) = \left(\frac{w_t - w_0}{w_0} \right) \times 100 \quad (3)$$

Where w_t is the coupon mass after time t . coated coupons were immersed up to 21 days.

3. Results And Discussions

3.1 Hardness

Figure 2 (a) and (b) shows the hardness of multilayers epoxy coating to steel substrate filled with Al₂O₃ microparticles and Al₂O₃ nanoparticles under dry and wet condition, respectively. In wet condition, the coated steel specimens were immersed in salt solution for 35 days. The improvement in hardness in dry and wet conditions was attained in both sizes of Al₂O₃ particles as compared to pure epoxy coating. Moreover, as the weight percentage of the micron and nano particles increases, the hardness increased. This increase in hardness is due increasing the Al₂O₃ particles content up to 3wt% in the surface of coated steel specimens may be attributed to the high intrinsic hardness of the Al₂O₃ particles itself. Furthermore, on the load applied through the hardness indenter, the compressive force increases which in turn presses the polymeric matrix, as a result, particles touch each other offering resistance. As the weight percent of Al₂O₃ particles content increases, particles fill in the gaps presented in the matrix thus forming a more dense structure so, hardness increases [17], [18]. In addition, the hardness of the stepwise graded micro/nanocomposite coating gives high hardness as compared to composite liner filled with 1wt% and 2wt% micro/nano Al₂O₃ particles. This may be attributed to the higher percentage of micro/nano Al₂O₃ particles (3wt%) in the outer surface of coated steel specimens, followed by 2wt% Al₂O₃ particles then 1wt% Al₂O₃ particles.

It can be depicted from Fig. 2 (a, b) that the hardness deteriorated as the coated specimens immersed in salt solution. This decreasing of the hardness value is mainly due to the seawater absorption which

produces plasticization, swelling and interfacial damages of the polymeric composite coating. Due to uptake of seawater molecules by the coated specimens, the connection between molecules of epoxy may disturb and the polymeric composite liners become so soft that the attachment between Al₂O₃ particles and epoxy got weakened. These combination phenomena reduce the material properties and also generate micro cracks inside the coated specimen that in turn weakens the hardness of the composite coating [19].

Figure 3 shows a comparison between the hardness values of microcomposite and nanocomposite coating under dry and wet conditions. It is clear from the figure that the highest hardness value was obtained with the addition of 3wt% nanometer-sized Al₂O₃ particles either in dry or wet condition. Followed by stepwise graded nanocomposite coating. This indicates the high effect of Al₂O₃ nanometer sized particles in strengthen the epoxy matrix. It is attributed to the great surface area of Al₂O₃ nanoparticles compared to Al₂O₃ microparticles [20]. The specimen N3 exhibited the highest hardness value with an improvement of 48.4% and 90.48% in dry and wet condition, respectively.

3.2 Tensile properties

The tensile strength of multilayers epoxy coating to steel substrate filled with Al₂O₃ microparticles and Al₂O₃ nanoparticles under dry and wet conditions is shown in Fig. 4 (a, b), respectively. As the weight percentage of the micron and nano Al₂O₃ particles increased, the tensile strength decreased. Adding 1wt% micro/nano Al₂O₃ particles gives the maximum enhancement of tensile strength in dry and wet conditions as compared to pure epoxy coating. Incorporation of small weight percentages of fillers leads to substantial improvement in mechanical properties to polymeric composite [21]. From Fig. 4 (a), it is clear that when adding the fillers with micron size to the epoxy with different weight percentages, the tensile strength was enhanced. The tensile strength of M1 was close to M123 that exhibited an improvement of 5.97% as compared to pure epoxy coating in dry condition. The specimen M1 exhibited the highest improvement of 2.31% under wet conditions. However, the least improvement of 0.66% and 0.92% in tensile strength was obtained with the specimen M3 under dry and wet condition, respectively. Figure 4 (b) shows that the specimen N1 exhibited the maximum improvement in tensile strength of 6.92% and 4.33% under dry and wet condition, respectively. The least improvement was obtained with the specimen N3. The addition of higher nanofiller weight percentage implies worse dispersions. The aggregations generally act as stress concentrator which in turn decreasing the mechanical properties [22–27]

Figure 5 shows a comparison between the tensile strength of microcomposite and nanocomposite coating under dry and wet conditions. The figure shows that the tensile strength was deteriorated as the coated specimens were immersed in salt solution. The water absorption primarily induces plasticization, decreasing the mechanical strength and rigidity of composite materials. At the beginning, the water absorbed interacts chemically with epoxy through Van der Waals force and hydrogen bonds, causing increased chain segment mobility. Water absorbed induces plasticization and deteriorates the mechanical strength of the composites [22]. The specimen N1 exhibited the maximum improvement in tensile strength under dry and wet condition, respectively. The least improvement was obtained with the

specimen M3. Both the stepwise graded steel lined with micro/nanocomposite are close to specimens coated with epoxy filled with 1wt% Al₂O₃ micro/nanoparticles.

Figure 6 (a, b) shows the tensile strain of multilayers epoxy coating to steel substrate filled with Al₂O₃ microparticles and nanoparticles. When adding the Al₂O₃ micro/nanoparticles to the epoxy, the tensile strain improved as compared to pure epoxy under dry and wet conditions. As the weight percentage of Al₂O₃ fillers increases, the tensile strain increases. Immersing the specimens in a salt solution leads to increasing the tensile strain of both sizes of Al₂O₃ particles as compared to pure epoxy coating. The ductility for both unfilled epoxy and Al₂O₃ filled micro/nanocomposite was enhanced as a result of water absorption. This can be attributed to the plasticization effect of water as the immersion time increased which can improve the ductility of the epoxy resin [28–30]. Figure 7 shows a comparison between the tensile strain of micro/nanocomposite coating under dry and wet conditions. The maximum improvement in in tensile strain in the dry condition and wet condition is obtained from N123 by 37.15% and 35.5, respectively. This is followed by an enhancement of 23.4% and 30% with N3 specimen in dry and wet conditions as compared to pure epoxy, respectively.

3.3 Barrier properties

Figure 8 (a, b) shows the barrier properties of multilayers epoxy coating to steel substrate filled with Al₂O₃ micro/nanoparticles immersed in salt solution and citric acid for 35 days. It is clear from the figures that the uptake% is significantly higher than that of sea water for both Al₂O₃ micro and nanocomposite coatings.

Figure 9 (a, b) shows the barrier properties of multilayers epoxy coating to steel substrate filled with Al₂O₃ micro/nanoparticles. The uptake for both solutions was decreasing with decreasing the size of the Al₂O₃ particles. Nanoparticles decreased the water permeability of the coating and enhanced its resistance against hydrolytic degradation [12]. Improved corrosion and mechanical properties were observed by filling the cracks presented in epoxy coats. The nanoparticles act as a strong barrier which can avoid penetration of aggressive ions to the steel surface [2]. Consequently, nanoparticles with very fine grain size and high boundary volume offer enhanced barrier properties as compared to conventional fillers [9].

The least uptake % was attained with steel coated with 1wt% Al₂O₃ nanoparticles. This can be attributed to the good dispersion of low weight percentage (1wt%) of Al₂O₃ nanoparticles as shown in Fig. 10 (a). Better properties can be accomplished when good dispersion and distribution of nanofillers are attained in the polymeric composites [31]. Inclusion of small weight percentages of nanofillers indicate substantial improvement in properties [21], [32].

It is observed that further increase in Al₂O₃ micro/nanoparticles to the epoxy, the uptake percentage increased. This may be attributed to the presence of agglomeration caused by the addition of more Al₂O₃ particles to epoxy that helps in more absorption of water as shown in Fig. 10 (b). Therefore, the bigger the free volume of cured resin and the clearance between Al₂O₃ particles and epoxy resin, the more water permeable for the micro/nanocomposites are and the worse barrier properties can be attained.

Furthermore, extra free volume presented at the interface also assisted the water permeability inside micro/nanocomposites. Sea water is more likely to diffuse along the epoxy/ Al_2O_3 interface and destroy the interfacial bonding rather than diffuse through the epoxy matrix. So, as the free volume increased, water permeability increased [33]. Aggregation possesses rise to lower surface interactions of Al_2O_3 -epoxy and higher stress concentration. This lead to lower the mechanical and barrier properties of the composites filled with nanofillers. However, smaller aggregate size results in highly improved mechanical properties [34].

The stepwise graded nanocomposite coating (N123) possess good barrier and mechanical properties. In order to determine the distribution of Al_2O_3 nanoparticles in the composites, surface analysis of the coated specimen N123 was done by FESEM and the composition scanning (EDX) images shown in Fig. 11. The FESEM was conducted on the examined nanocomposite coating on which the surface scanning was performed, and EDX provides the results of the examination of the coated steel N123. The findings of surface scanning show a homogeneous distribution of elements in the structure.

4. Conclusions

In this work, the mechanical and barrier properties of steel lined with multilayered epoxy filled with Al_2O_3 particles in micron and nano size was investigated. The results showed that the tensile properties, hardness and epoxy coating barrier resistance against salt solution and cetric acid media were significantly enhanced using nano and micro sized Al_2O_3 particles. Mechanical and barrier properties of the nanocomposite coating was substantially higher than microcomposite coatings. The steel lined with multilayered epoxy filled with 3 wt% Al_2O_3 nanoparticles exhibited the highest hardness value with an improvement of 48.4% and 90.48% in dry and wet conditions, respectively. However, the steel lined with multilayered epoxy filled with 1 wt% Al_2O_3 nanoparticles had the highest enhancement in tensile strength of 6.92% and 4.33% under dry and seawater conditions, respectively. Moreover, the maximum improvement in tensile strain in the dry and seawater conditions was obtained from stepwise graded nanocomposite coatings by 37.15% and 35.5, respectively. It is observed that further increase in Al_2O_3 micro/nanoparticles to the epoxy coating, the uptake percentage of salt and cetric acid solution increased. The least uptake percentage was attained with steel coated with 1wt% Al_2O_3 nanoparticles.

Declarations

Conflicts of interest

The authors declare no conflict of interest.

Data availability statement

The datasets used during the current study available from the corresponding author on reasonable request.

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Figures

Figure 1

Construction of micro/nanocomposite coatings on steel substrate

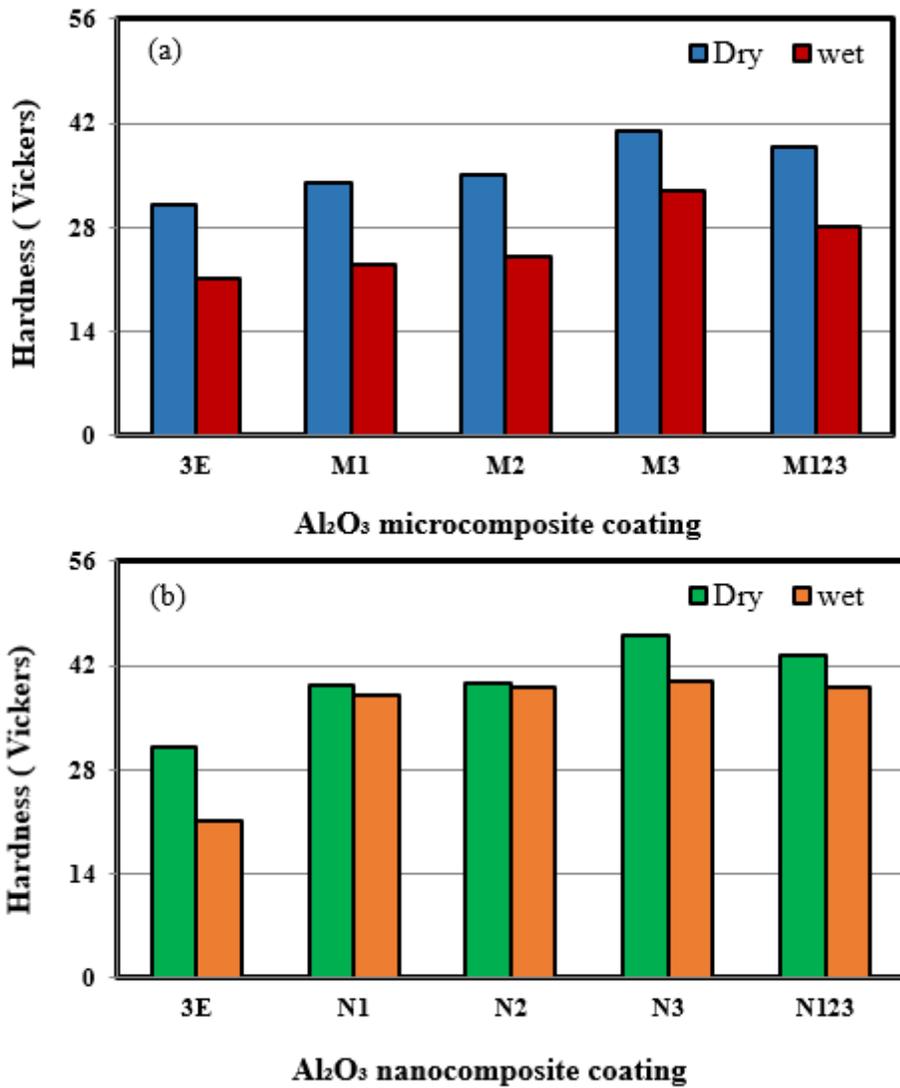


Figure 2

The hardness of multilayers epoxy coating to steel substrate filled with

(a) Al_2O_3 microparticles (b) Al_2O_3 nanoparticles

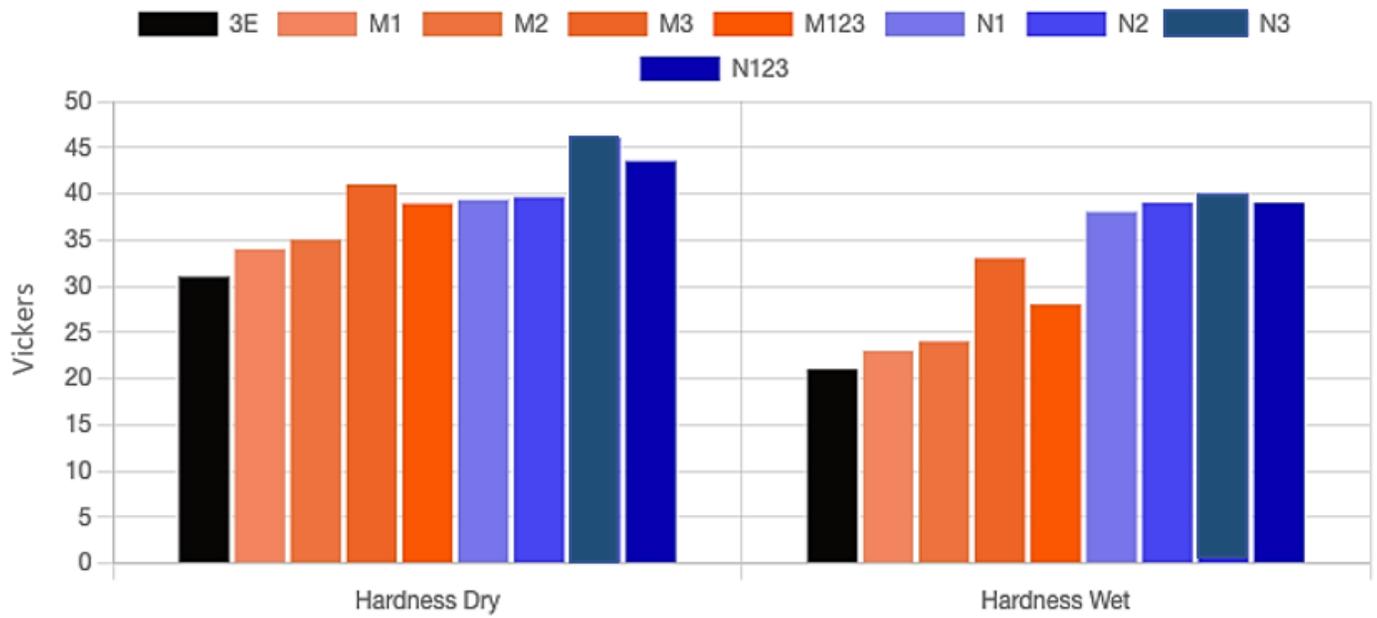


Figure 3

A comparison between the hardness values of microcomposite and nanocomposite coating under dry and wet conditions

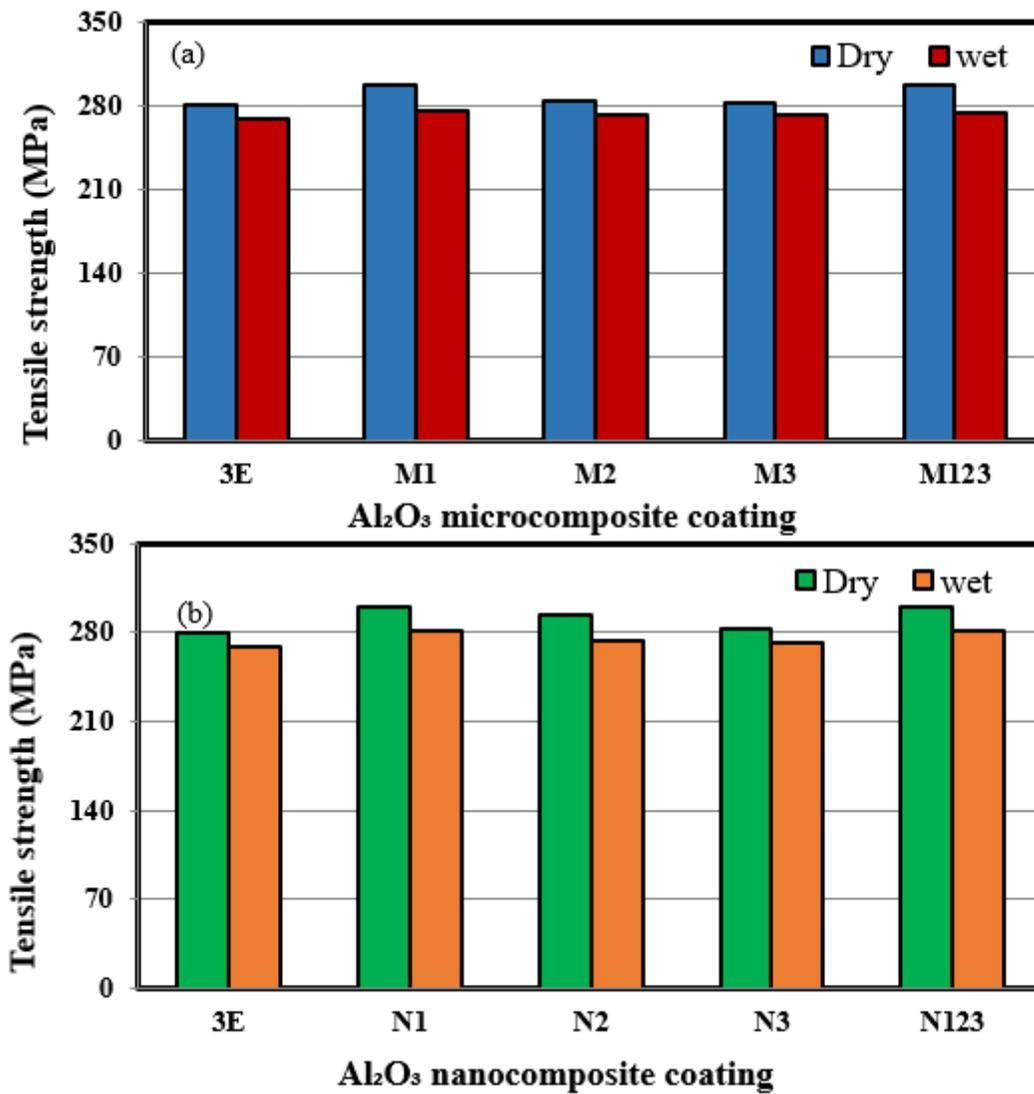


Figure 4

The tensile strength of multilayers epoxy coating to steel substrate filled with

(a) Al_2O_3 microparticles (b) Al_2O_3 nanoparticles

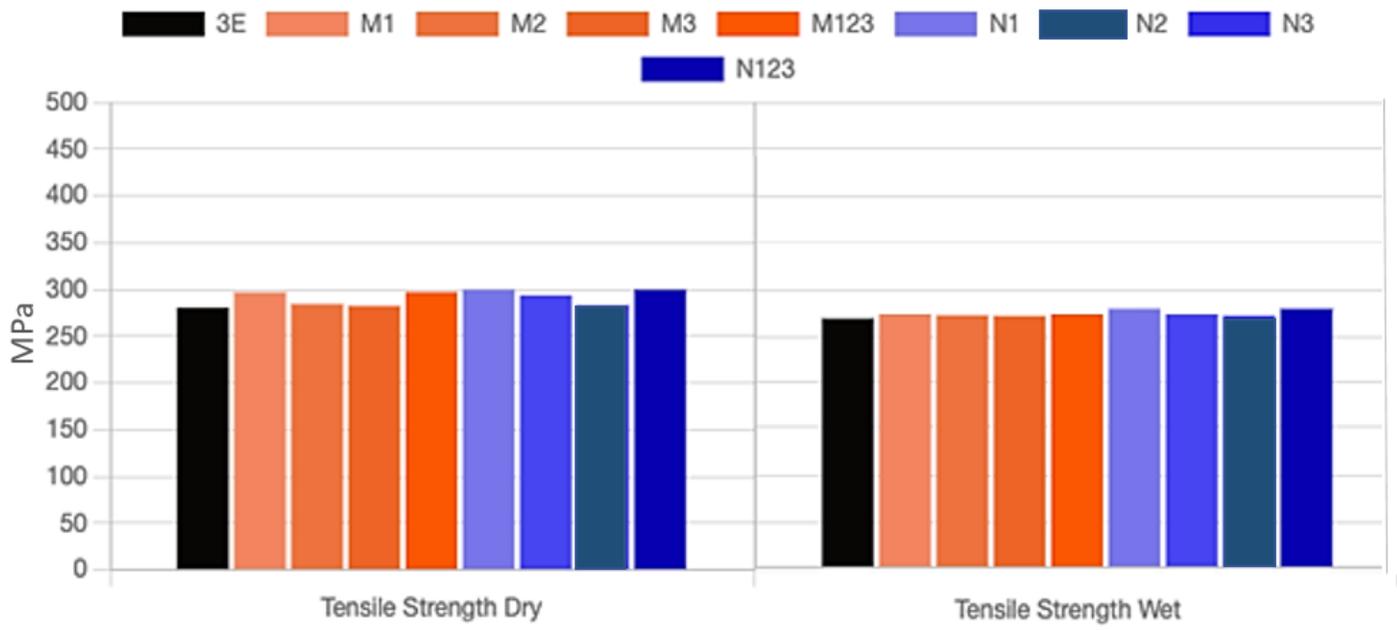


Figure 5

Tensile strength of micro/nanocomposite coating under dry and wet conditions

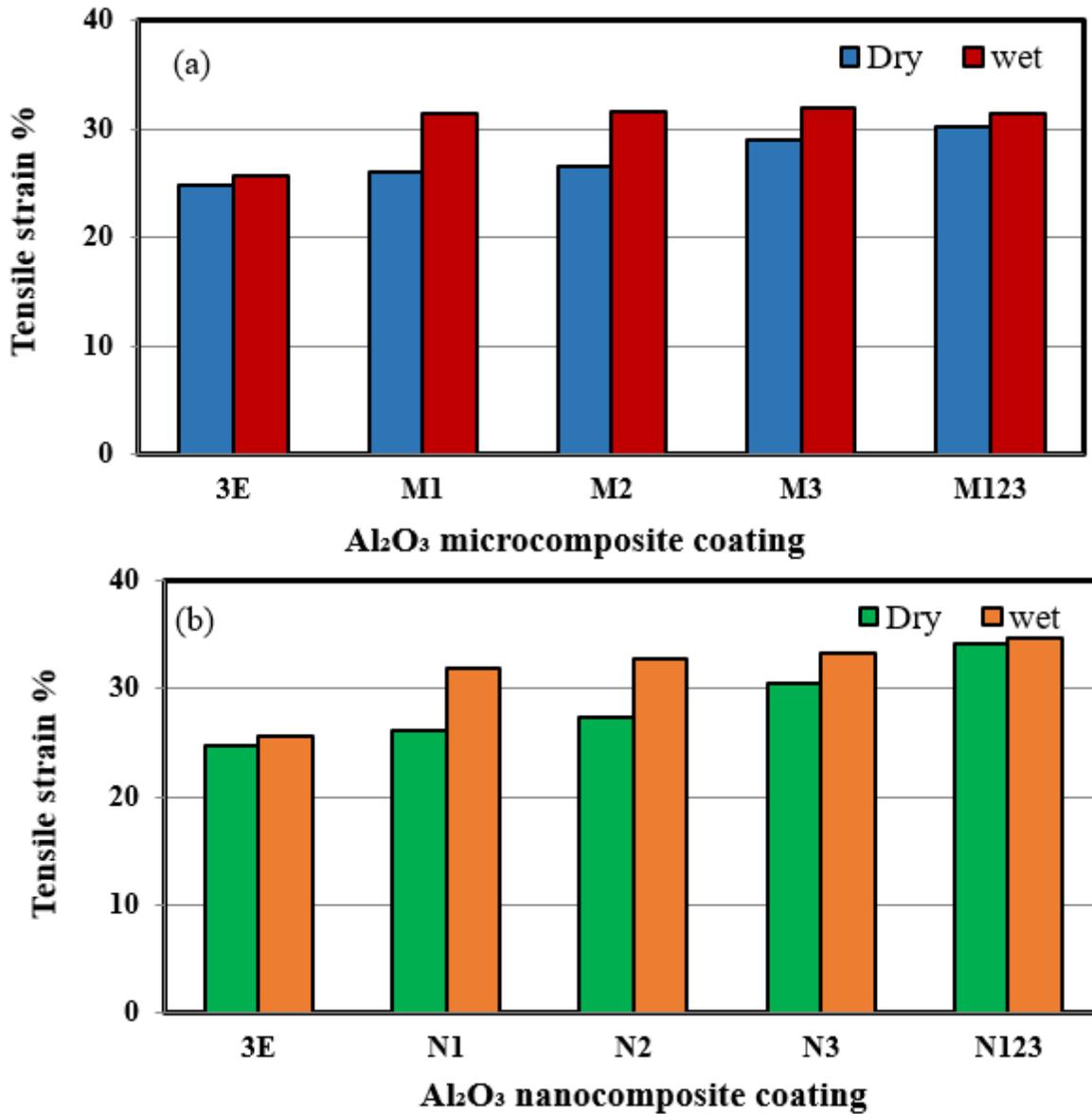


Figure 6

The tensile strain of multilayers epoxy coating to steel substrate filled with

(a) Al_2O_3 microparticles (b) Al_2O_3 nanoparticles

Figure 7

Tensile strain of micro/nanocomposite coating under dry and wet conditions

Figure 8

The barrier properties of multilayers epoxy coating to steel substrate filled with Al_2O_3 micro/nanoparticles immersed for 35 days in (a) salt solution (b) citric acid

Figure 9

The barrier properties of multilayers epoxy coating to steel substrate filled with Al_2O_3 microparticles (a) Al_2O_3 nanoparticles

Figure 10

SEM showing the dispersion of alumina nanoparticles in (a) N1 and (b) N3

Figure 11

(a) FE-SEM of stepwise graded nanocomposite coating N123, (b)-(d) elemental map, (e) EDX spectrum