Surface Roughness of ZnO-SiO$_2$ Nanocoating

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Abstract. Nanocoating acted as a barrier to avoid the transportation of corrosive species. Nanoparticles provide the resin or coating with a continuous, solid and protective network layer. Silicon wafer p-type substrate $<101>$ was used as standard materials while tungsten wire of 0.15mm diameter was used as tip for scanning operation. Nanoeducator showed that present of nanoparticles increase the surface roughness and maintain the size of nanoparticles. Nanoparticles well distribute in nanosize. Immersion test showed nanoparticles also, improved the adherence of the cured epoxy coating.

1. Introduction

Nowadays, most of developing countries have put more interest and giving attention to nanotechnology areas because it gives advantages in terms of environmental concern, military, industrial and research area. Several reasons for surface of materials to be coated are due to biocompatibility which is reported to increase life time, wear protection, thermal and mechanical stability and at the same time reducing friction and corrosion effect [1]. According to latest study by Nasional Association of Corrosion Engineer (NACE), the global cost of corrosion is estimated to be $2.5 trillion equivalent to roughly 3.4 percent of the global Gross Domestic (GDP). By controlling the corrosion, it is possible to save around $375 to $875 billion annually on a global basis and save the environment from corrosion affect [2]. Corrosion is always the major of energy saving and material loss. The problem is due to overcome corrosion problem. Corrosion is gradual degradation process of metal through chemical reaction. Mostly happen by electrochemical and reaction with the surrounding environment. It affects material properties [3].

In this research, Nanoeducator as scanning probe microscope (SPM) has been used to study the surface structure up to atomic scale and study the compability of coating. The surface roughness and nanoparticle size of nanocoating determined using this method.

2. Methodology

Nanoeducator was used for studying the surface properties of materials and substrate. In this research, silicon wafer p-type substrate $<101>$ was used as standard materials while tungsten wire of 0.15mm diameter was used as tip for scanning operation. Since in the instruction manual book has listed down the suitable length for the tip preparation of tungsten wire which is 18.5 ± 0.3mm (max), it is advisable to make around 12.0 mm. Silicon wafer was cut into small fragment using diamond cutter. Then, ultrasonic agitator cleaned it using acetone for 15 minutes. After cleaning, it was dried. The coating sample under silicon wafer as substrate was prepared. After tip and sample had been prepared, tip was inserted into probe while the silicon wafer was mounted on a magnetic holding plate using tape. Designation of sample used as stated in table 1. Table 1 also shows nanocoating with certain wt% of nanoparticles. Nanocoating was also observed under salt spray testing machine from day one until 60 days.
Table 1. Weight percentages of nanoparticles loading in nanocoating.

| Designation | Nanoparticles (wt%) |
|-------------|---------------------|
| 1Z4S        | 1ZnO+4SiO₂          |
| 2Z3S        | 2ZnO+2SiO₂          |
| 3Z2S        | 3ZnO+2SiO₂          |
| 4Z1S        | 4ZnO+1SiO₂          |

3. Results and Discussion

Top view images of three-dimensional (3D) were taken in order to surface topology evaluation of the silicon wafer, epoxy coating and ZnO/SiO₂ coating was presented in Figure 1. The images were recorded at 10000×10000 nm planar in tapping mode and velocity at 1000 nm/s. Obviously, the surface of the uncoated and coated specimens with nanoparticles ZnO and SiO₂ was different. Evidently, the obtained coatings are more homogeneous and the scratch of specimens was not visible. The uniform allocation of single and non-agglomerated ZnO/SiO₂ coating was also revealed by nanoeducator. Not only surface region analysis was performed but also surface roughness was estimated qualitatively by evaluating the SPM pictures.

Figure 1 shows that neat sample without any nanoparticles completely showed heterogeneous and change in surface roughness compared to sample containing ZnO/SiO₂ coating. It was due to breakdown of polymer matrix due to long exposure to the electrolyte. Sample containing nanoparticles showed only a little changes in on the surface, indicates better protection for substrate.

![3D Image of Silicon Wafer](image1)

![3D Image of epoxy coating](image2)

![3D Image of ZnO/SiO₂ coating](image3)

Figure 1. Nanoeducator topography (3D) of the surface. a) silicon wafer; b) epoxy coating and c) ZnO/SiO₂ coating

Figure 2 shows roughness of the surface of the silicon wafer, epoxy coating and ZnO/SiO₂ coating. The silicon wafer, epoxy coating and ZnO/SiO₂ coating was observed as evenly distributed higher
peaks on the layer surface leading to average roughness of approximately 90 nm-100 nm. It can be also observed that the coated samples with nanoparticles had nano roughness on the surfaces. It is interesting to note, that addition of nanoparticles into coating did not affect the size of particles on the surfaces. Thus, according to the nanoeducator measurements, it can be concluded that even coatings of nanoparticles into epoxy on the silicon wafer as substrate showed the size still remain in nano size.

![Figure 2](image)

**Figure 2.** Nanoeducator roughness of the surface. a) silicon wafer; b) epoxy coating and c) ZnO/SiO$_2$ coating

**Table 2.** Nanoeducator roughness of the surface of the silicon wafer, epoxy coating and ZnO/SiO$_2$ coating

| Test                  | Silicon wafer | Epoxy coating | ZnO/SiO$_2$ coating |
|-----------------------|---------------|---------------|---------------------|
| Surface roughness (nm)| 92.58         | 118.31        | 91.82               |

Table 2 represents surface roughness values for silicon wafer, neat epoxy coating and ZnO/SiO$_2$ coating. It was found that the surface roughness of neat sample is higher due to breakdown of polymer matrix as compared to the surface roughness of ZnO/SiO$_2$ coating [4]. This suggests the improvement in the properties of the coating after modification. The surface roughness of coating modified with ZnO/SiO$_2$ was relatively increased than the coating modified with epoxy coating. It was confirming that loading of nanoparticles into the resin is sufficient to fill all inter pigmentary interstices leading to increase the corrosion resistance [5].

Figures 3 shows appearance of nanocoating after immersed in NaCl from day one until 60 days. Based on images of nanocoating, all samples sustain to suffer from corrosion. However, the best image was nanocoating with 3wt% ZnO and 2wt% SiO$_2$. The reason is that, the nanoparticles was distributed uniformly into matrix (epoxy) and having good bonding process. Nanocoating acted as a barrier to avoid the transportation of corrosive species, such as chlorine and hydroxyl ions, water,
oxygen, pollutants and pigments. They can absorb more resins compared to conventional pigments and thus reduce the free space between the pigment and the resin. Thus, nanoparticles increase the transport path of corrosive species and enhance the protective properties and performance [6,7]. The confirmation was done by calculated the weight loss and corrosion rate for each sample.

Figure 3. Images of nanocoatings in 60 days (salt immersion) a) 1Z4S; b) 2Z3S; c) 3Z2S; d) 4Z1S; e) unpaint

Based on the results obtained, corrosion certainly occurred since weight loss was evident. Neat epoxy (0wt% ZnO) samples showed the greatest weight loss regardless of the type of media it was immersed in. The 3wt% ZnO and 2wt% SiO$_2$ recorded the smallest weight loss that is 62 mg with 3wt% ZnO. It was supported by Shambu Sharan Kumar (2015) studies, the corrosion protection efficiency has been improved when nanoparticles have been incorporated and dispersed into coating.

Table 3 shows the weight loss data and corrosion rate of mild steel in water and NaCl, respectively. The corrosion rate was calculated using the formula described previously. From the data, it was found that the epoxy coating with 3wt% ZnO composition showed the smallest weight loss and consequently the smallest corrosion rate value in water, having a value of 0.00073 mmpy. Nanoparticles improved the quality of the cured epoxy coating and improved barrier performance of the epoxy coating. Nanoparticles also, improved the adherence of the cured epoxy coating. Addition of nanoparticles into coatings delays the corrosion process. Nanocoating acting as an effective barrier in preventing corrosion from happen. Nanocoating was reported to increase lifetime, wear protection, thermal and mechanical stability and at the same time reducing friction and corrosion effect [8,9]. The weight loss was calculated as shown in Table 3.

| Wt% of nanoparticles | Weight loss (mg) in NaCl | Corrosion rate (mmpy) in NaCl |
|-----------------------|--------------------------|-----------------------------|
| 0                     | 880                      | 0.1029                      |
| 1Z4S                  | 181                      | 0.0212                      |
| 2Z3S                  | 123                      | 0.0144                      |
| 3Z2S                  | 62                       | 0.0073                      |
| 4Z1S                  | 126                      | 0.0147                      |
4. Conclusion
As summary, presence of nanoparticles increases the surface roughness. Nanoparticles contribute as curing agent towards epoxy. It was because nanoparticles can interlock in between the polymer structure. Nanoparticles well distribute in nanosize in nanocoating.

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