Synthesis and Characterization of Low-Cost Epoxy-Based Erosion Resistant Nanocomposite Coating

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Abstract: We report a simple route to synthesized erosion resistant epoxy-based nanocomposite coatings. The silica nanoparticles were surfaced modified using stearic acid and then incorporated into the epoxy coating. The resulting nanocomposite coating films were characterized for erosion resistance, mechanical and thermal stability. For the application on turbine blades, conventional techniques were used. It was found that for the incorporation of nano silica into the epoxy matrix, surface modification was essential. Besides, incorporation of silica resulted in considerable improvement in the resistance to erosive wear and a life span improvement of around 36 percent was achieved. Similar trend was observed for the Shore D hardness which increases from 60 for the virgin coating to 70 for the nanocomposite coating.

Keywords: Polymeric nanocomposite, epoxy coatings, silica nanoparticles, erosion resistance.

INTRODUCTION

Erosion of turbine blades is a common engineering problem which results in high cost of repair and replacement [1, 2]. One possible solution to this problem is to develop efficient erosion resistant surface coating systems [3]. Polymer-inorganic nanocomposites materials are considered as the materials of the 21st century owing to their significantly improved properties compared to pristine materials [4]. They offer better hardness, enhanced erosion and scratch resistance, excellent mechanical strength, and superior thermal and optical properties [4-6]. Owing to these features, nanocomposites have gained significant attention in the field of coating technology [3]. The enhanced properties they offer are very important and highly desirable in strong, erosion resistant next generation coatings formulations [3].

In this paper, we report a simple approach to incorporate inorganic nanofiller silica in the epoxy-based coating system generally employed on turbine blades. Use of silica as a micron and nanoscale filler to improve properties of a wide range nanocomposite materials is well established [3]. These nanofillers, if incorporated into the polymer matrix at maximum exfoliation, are known to enhance mechanical properties of resulting nanocomposite coatings [5, 6]. However, as the silica nanoparticles are naturally hydrophilic, their incorporation into the polymer matrix would require suitable surface modification approach enabling their complete dispersion into the epoxy matrix. Various approaches are reported in literature for the surface modification of fillers. One such approach is the physical adsorption of organic surfactants onto the filler surface making them hydrophobic and hence compatible with the polymer matrix [7]. Another approach is based on the use of conventional coupling agents [8].

In this work we explored Stearic Acid as a *nonreactive* modifier [9]. The prepared nanocomposite epoxy coatings were characterized for erosion resistance, mechanical and thermal stability. For the application on the turbine blades, conventional coating techniques were used. Our results demonstrate the successful synthesis of silica-reinforced epoxy coatings which exhibit considerably improved properties compared to pristine epoxy coatings. The developed coatings showed improve resistance to erosive wear, enhanced hardness and better thermal stability. We believe that the developed nanocomposite epoxy coatings will likely find applications as specialty coatings such as in aerospace applications and as hi-tech metal coatings.

EXPERIMENTAL

Materials

Fumed Silica (Average particle diameter: 12 nm, purity > 99.8%, specific surface area: 200m2/g, MP:

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1610°C, specific gravity: 2.2) was used as received. For surface modification, the granular type Stearic Acid obtained from Sigma Aldrich (Mol. wt: 284.48, MP: 68 – 71°C, BP: 361°C, wetting surface of the Stearic Acid: 445 m^2/g) was used as received. Ethyl Acetate (Purity > 99.5 %) was obtained from Sigma Aldrich and used without further purification. The epoxy resin used in this study was liquid diglycidyl ether of bisphenol A (DGEBA), Ep. 5.2-5.5: Epoxide equivalent of 182-192, viscosity of 11000-13000 mPa s (at 25 °C). For the application of coatings, AISI 1020 low Carbon steel was used as substrate.

Preparation of Erosion Resistant Polymer-Nanocomposite Coating

First, surface modification of silica nanoparticles was performed using stearic acid as non reactive modifier. The carbonyl oxygen of the stearic acid molecule can form hydrogen bonds with the silanol hydrogen enabling them to adsorb on the silica surface [10] (Scheme **1**).

Stearic acid was added to ethyl acetate and the resulting mixture was stirred for 1 h, followed by the gradual addition of fumed silica nanoparticles. The mixture was stirred for another hour. The resulting dispersion was placed in a fume hood under a constant air flow to allow removal of solvent. Washing of the resulting modified silica particles was done by ethyl acetate for 30 minutes followed by filtration to remove the excess stearic acid.

The recipes used for the synthesis of silica based erosion resistant nanocomposite coatings are summarized in Table **1**. Briefly, different amounts of modified nanosilica particles (5, 10, 15, 20 & 25 wt. %, based on the amount of DGEBA) were added to the coating formulation. Firstly, known amount of epoxy was added in a beaker. Modified nanosilica particles were dispersed separately in a flask in 40 ml of ethyl acetate. The dispersion was then added to the epoxy under stirring. The resulting mixture containing DGEBA epoxy and modified nanosilica particles was mixed for around 30 min at 50°C using a mechanical agitator (300 rpm) resulting in a homogeneous and uniform mixture. The mixture was allowed to cool to room temperature followed by the addition of stoichiometric amount of the curing agent and mixing for another 30 minutes. The resulting coating was then applied on a sample placed in a mold of volume (75mm x 25mm x 4.5 mm).
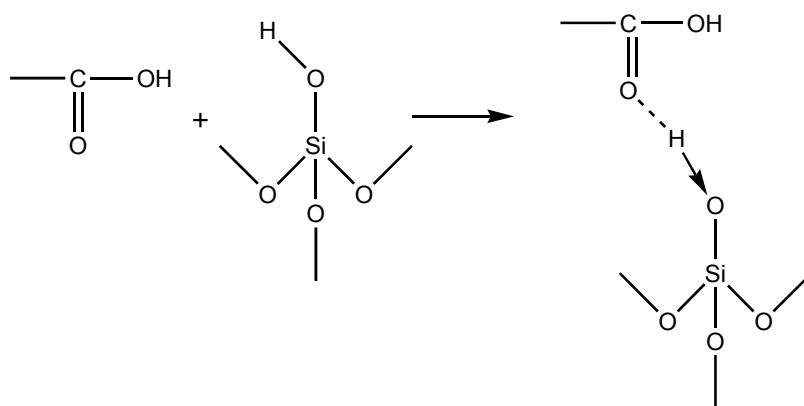
Scheme 1: Surface modification of silica [10].

Table 1:   Recipes for the Synthesis of Erosion Resistant Nanocomposite Coatings

Sample Code	Silica wt. %	Epoxy hardener ratio	Curing time (days)
SP-1	0	3:1	7
SP-2	5	3:1	7
SP-3	10	3:1	7
SP-4	15	3:1	7
SP-5	20	3:1	7
SP-6	25	3:1	7

Characterizations

The surface modification of the silica nanoparticles was investigated by using thermogravimetric analysis (TGA). The erosion resistance of the coating was determined by erosion testing machine according to the procedure described in ASTM G76 standard. Briefly, a sixteen grit silicon carbide particulate was directed at the surfaces of the coated samples at a rate of 2.46 kg per minute by a high pressure air nozzle with a diameter 8.3 mm. The nozzle was positioned 24 inches from the coatings and the stream of particles directed toward the coating surfaces by a 71.07 meter per second air stream at an angle of 90 degrees. Furthermore, the Shore D Hardness was measured on a manually operated Durometer equipped with an indenter that is attached to a graduated (0 to 100 units) digital scale. The samples were measured following the procedure described in the ASTM D2240 and the results were averaged for four independent measurements taken from different parts of the samples.

RESULTS AND DISCUSSION

The weight loss curves for pristine silica (SiO_2) and stearic acid-modified silica ($C_{18}H_{36}O_2$–SiO_2) as a function of temperature are shown in Figure **1**. Since silica is resistant to high temperature, no degradation was expected within the temperature range explored (20 to 600 ºC). The nonreactive modifier $C_{18}H_{36}O_2$ however, is expected to burn off all whilst retaining the silica. As shown in Figure **1**, for the pristine silica, a weight loss of around 3 wt % was observed between 30 °C and 150 °C until a plateau was reached. This
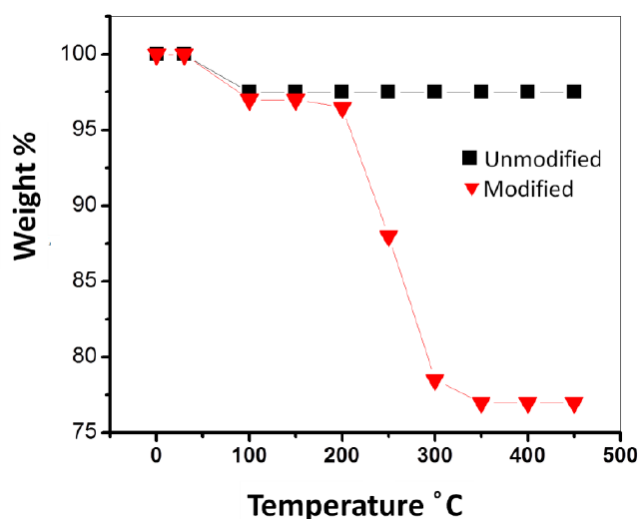
initial weigh loss corresponds to the evaporation of the moisture content present in silica particles. For the modified silica ($C_{18}H_{36}O_2$–SiO_2) sample, two main weight loss stages were observed. First stage was observed between 30 °C and 150 °C which correspond to the moisture content loss. The second stage ranges from 200 °C to 400 °C where the decomposition of surface stearic acid resulted in considerable weight loss.

The formulated coatings containing different amounts of silica were applied on a metal substrate followed by curing which resulted in a strong, smooth epoxy coating (Figure **2**). The erosion resistance of the coatings were then determined by erosion testing machine according to ASTM G76 standard.
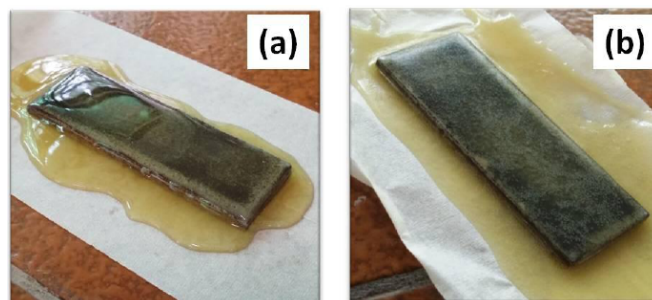

Figure 2: Application of nanocomposite coating on AISI 1020 steel substrate (**a**) before and (**b**) after curing.

Under the test conditions, as shown in Figure **3**, the virgin epoxy coating showed an erosion rate of 0.34 grams per minute. The nanosilica modified epoxy coatings showed a considerable improvement in the erosion resistance, which increases with the amount of filler being used. For the filler content of 5 wt% an erosion rate of 0.28 grams per minute was observed. Similarly erosion rates for 10, 15, 20 and 25 wt%
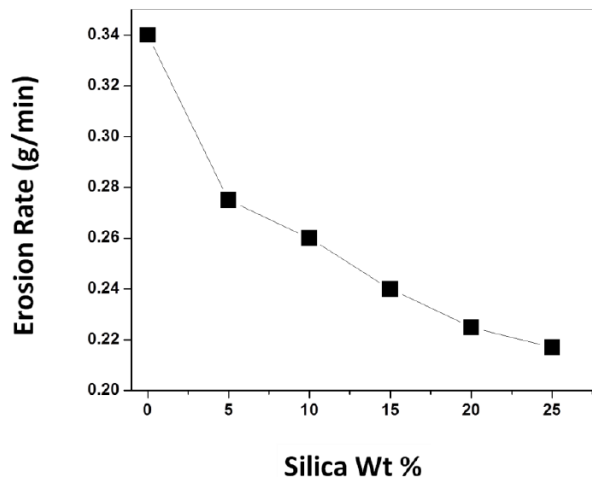

Figure 1: Thermogravimetric Analysis of Silica Modified with Stearic Acid.

Figure 3: Erosion rate (g/min) as a function of Silica wt%.

nanosilica incorporated epoxy coatings were recorded to be 0.26 grams per minute, 0.24 grams per minute, 0.22 grams per minute and 0.20 grams per minute, respectively.

Figure **4** shows the % life span improvement of the nanocomposite coatings as a function of nanofiller content. An improvement of more than 19 % in the coating life was observed just after the addition of 5 wt% silica. An overall improvement of 36% was observed compared to the erosion rate of virgin epoxy. The observed improvement in the erosion resistance can be explained using the mechanism described by Zhou *et al.* [11]. As the high velocity particles strike to the surface of the nanocomposite coating, they tear apart the matrix material. This exposes the incorporated silica particles which resist the impact of the colliding particles much better than the matrix due to their high hardness thus protecting the matrix underneath in the impact direction.
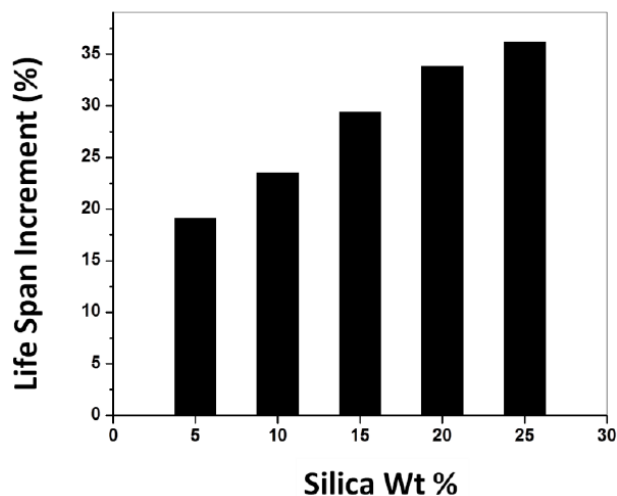

Figure 4: Life span improvement of the nanocomposite coatings as a function of silica content.

Shore D hardness was used to measure the surface hardness of the pristine and nanocomposite coatings. Figure **5** shows the effect of the silica content on the hardness of the coatings. The hardness of the pristine epoxy coating was measured to be around 60. This value decreases to 43 with the addition of 5 wt % and then showed an increasing trend with the increasing amounts of nanosilica. The initial decrease in the hardness for small amount of filler content is likely caused by the fact that the hardness is dependent on the intermolecular forces and compactness of the material. Addition of small amounts of filler interferes with the compactness and intermolecular attraction of epoxy. On further increasing the filler content the

hardness increases because of the dominating properties of nanosilica which has considerably higher hardness compared to the epoxy matrix.
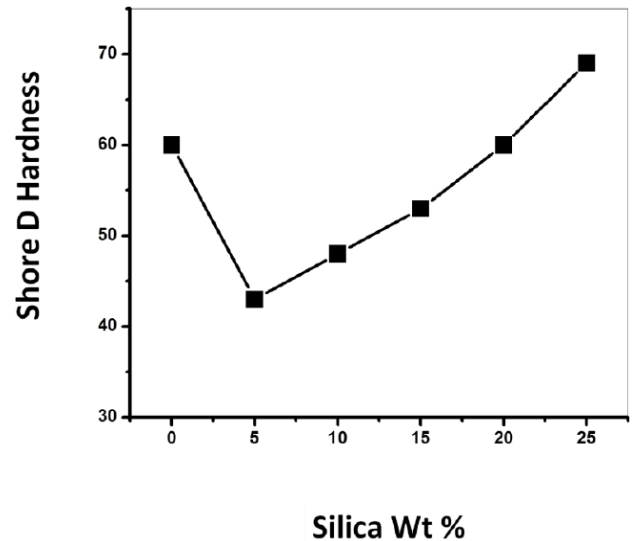

Figure 5: Hardness of the pristine and nanocomposite coatings as a function of silica content.

CONCLUSIONS

In this paper, a simple approach to synthesized erosion resistant epoxy-based nanocomposite coating using nanosilica as filler was presented. It was found that for the incorporation of nano silica in epoxy resin, surface modification of inorganic silica was essential. For this purpose stearic acid was found to be a low cost and effective non-reactive modifier. It was found that the resistance to erosive wear increases considerably as a function of nano silica content. An improvement of around 36% in the coating life span was achieved for the highest silica loading (25 wt %). Furthermore, the hardness of formulated coating also showed an increasing trend with the amount of filler being added.

From the results presented in this article, it can be concluded that nanosilica incorporated epoxy coating has a vast scope for erosion resistance in wind turbine blades and other applications used in erosive environment. In the future, this approach will be employed to synthesize high performance nanocomposite coatings using a variety of nanoparticles such as Alumina, zinc oxide, graphene and metals.

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