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Microstudies on thermal and UV treated silicone-acrylic paints with different pigment volume concentrations

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ABSTRACT The effect of pigment volume concentration (PVC) on morphology of pigments particles employed in the silicone–acrylic compositions suitable for high temperature corrosion protection application, has been investigated using SEM. Silicone-acrylic based paints with 50% silicone resin have been selected to prepare paints with PVC values from 10% to 60%. The paints were applied on mild steel panels of size of 5cm x 7.5cm. After curing for one week, the panels were subjected to 24 hours of heating at temperature ranging from room temperature up to 300°C and 400 hours of UV weathering. After the corrosion test, SEM micrographs were taken to investigate the gloss, rusting and adhesion properties of the coating films.

(Pigment volume concentration, Silicone-acrylic paint, Mild steel, SEM)

INTRODUCTION

Traditionally, coatings formulator worked with weight relationship, but volume relationships generally are of more fundamental importance and practical significance as reported by W.K.Asbeck and M.Van Loo [1]. The work considering the pigment volume concentration (PVC) has also been reported by other researchers [2,3].

Silicone resin has a very low surface energy, high thermal stability, UV transparent, and has a low glass transition temperature (T_g). These properties are utilized by the paint industry to provide excellent water repellency, resistance to weathering, UV radiation, and thermal cycling. Currently silicone resin based coatings are widely used in the design of heat resistant, corrosion resistant and weather resistant coatings [4,5].

Paints containing acrylic resin as binders have been known since the 1930s. They are one of the largest product classes in the paint and coating sector. It provides outstanding gloss and gloss retention, transparent and do not yellow even after prolong thermal stress [6].

In this work, paints containing a series of PVC values have been prepared from silicone-acrylic composition. This paper reports work using SEM to study the effect of UV and heating on the coating material.

EXPERIMENTAL SETUP

Sample Preparation

The composition of silicone-acrylic resin with a ratio of 50:50 has been selected to make corrosion resistant paint. The TiO_2 pigment and small quantity of additive were then added to the silicone-acrylic composition to make paints with different pigment volume concentrations (PVC) (10% to 60%) according to the formulation below:

$$\mathrm{PVC}(\%) = \frac{V_{P+A}}{V_{P+A} + V_B} \times 100$$

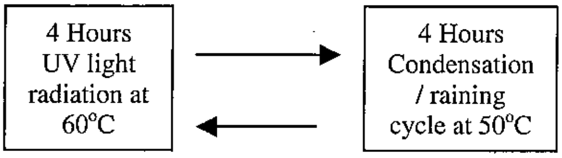
Where V_{P+A} is the volume of pigment and additive and V_B is the volume of solid binder. The mixture of pigment and resin were then ground in a ball mill with the help of porcelain or iron balls. The ingredients were ground and mixed well in the ball mill for 30 minutes to get the Hegman Gauge value of seven [7]. The paint was then coated on several mild steel panels of size 5cm x 7.5cm.

Heat resistant test

Each sample with different PVC values was subjected to temperatures from room temperature to 300°C for 24 hours in a muffled furnace. The samples were then taken out and left to cure at room temperature. These samples were observed by SEM to study morphology of the coating film.

UV weathering test

Weathering test was conducted using the QUV weathering tester model QUV/S. The weathering cycle is as shown below:



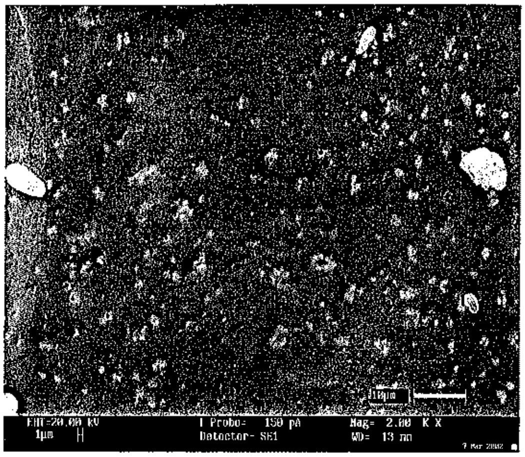
After 400 hours of prolonged UV radiation (270-390nm), temperature cycling and high humidity cycle, the samples were removed from the testing chamber and observed under SEM for any changes on gloss, rusting and adhesion failure of the coating films.

Scanning Electron Microscopy (SEM)

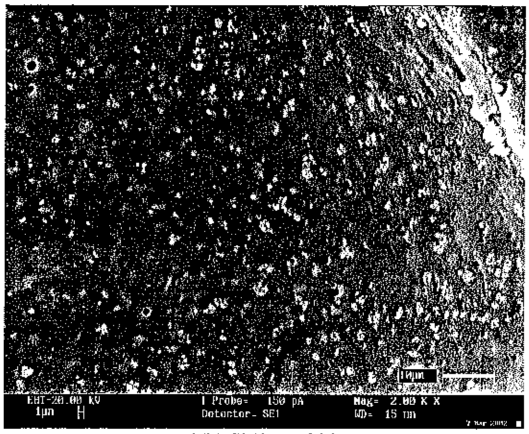
The samples were prepared by vacuum discharge coating with carbon to produce a conductive surface. A conductive tape was then applied to ensure good electrical contact between the specimen and the holder. The specimens were examined using a Hitachi-Amray scanning electron microscope, and suitable visual images were recorded photographically.

RESULTS AND DISCUSSION

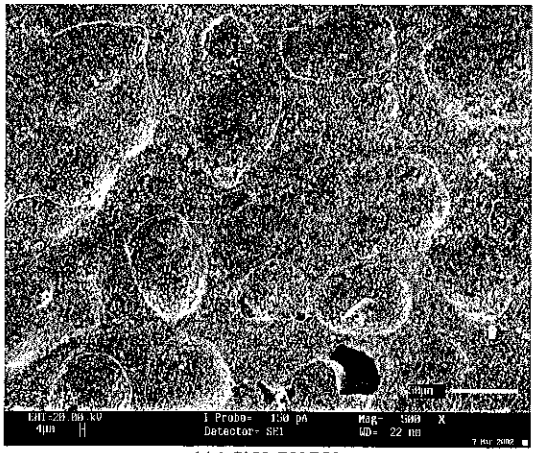
SEM for UV weathering test



1(a) Si50-PVC10



1(b) Si50-PVC30



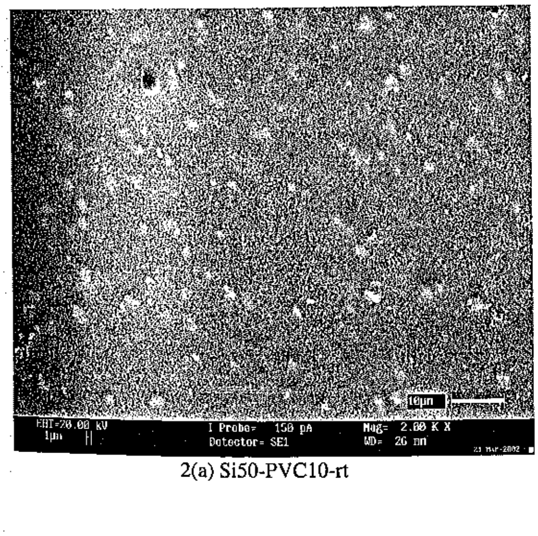
1(c) Si50-PVC50

Figure 1. Micrographs of Samples PVC (a) 10, (b) 30 and (c) 50 after 400 Hours of UV Weathering Test.

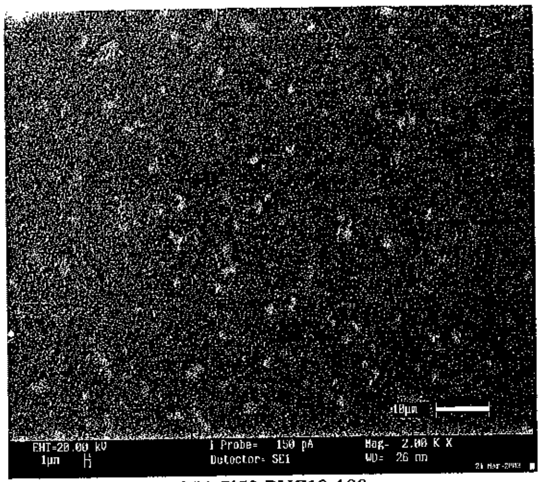
Figs.1(a), 1(b) and 1(c) showed micrographs for different PVC values. It can be observed that as the PVC value increased, the pigment particles were gradually exposed. This may due to the process of photo-degradation of small molecules such as xylene and resins which can either be evaporated or washed away by moisture [8].

For PVC10 as shown in Fig.1 (a), a thin brittle chalking surface layer has formed over a thicker more elastic layer [9]. This may due to the further loss of binder (resins) materials which effectively increases the PVC at the coating surface [10], followed by formation of brittle top chalking layer on the surface. However, there were no chalking observed on PVC30. For further increase in pigment volume concentration, cracks, chalking and yellowing can be observed at PVC50. This implies that the volume of pigment has exceeded volume of binder (resins) materials and was unable to hold the pigment particle on its location. Some of the pigment particles were exposed to the raining/heating cycle, and washed away by the erosion processes. This resulted in a brittle layer and exposure of the metal substrate as shown in Fig 1(c) [11-13].

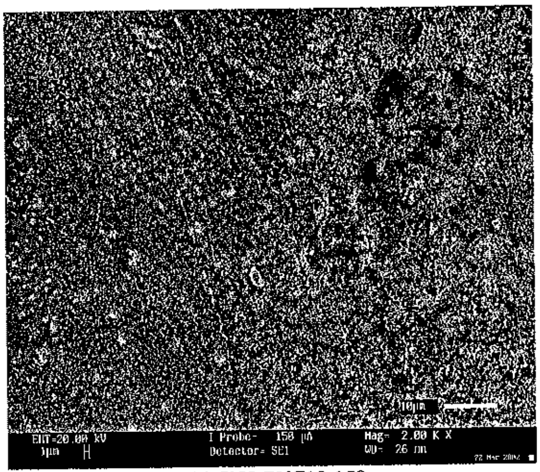
SEM for 24 hours heating test



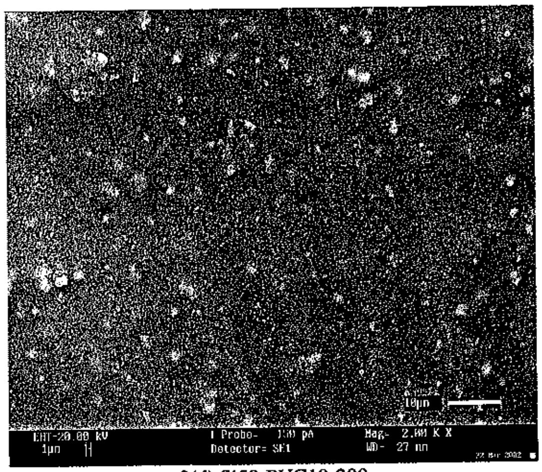
2(a) Si50-PVC10-rt



2(b) Si50-PVC10-100

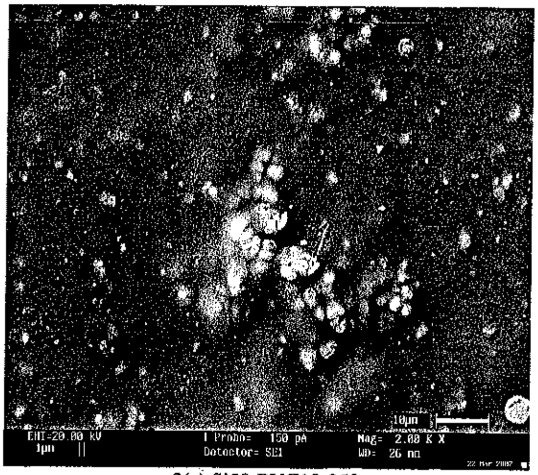


2(c) Si50-PVC10-150

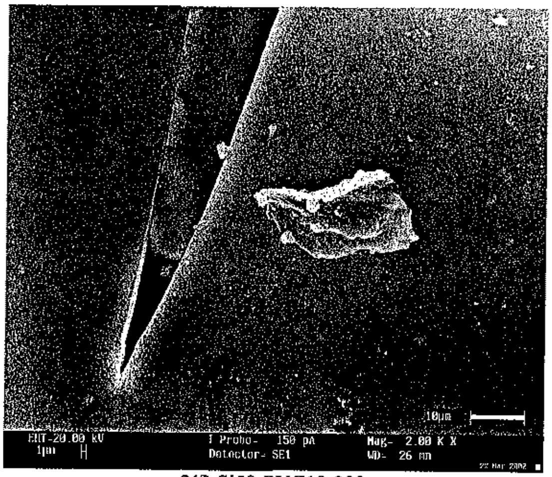


2(d) Si50-PVC10-200

Figure 2. Micrographs of Samples with PVC10 heated at (a) Room Temperature, (b) 100 °C, (c) 150 °C, (d) 200 °C, (e) 250 °C and (f) 300 °C.

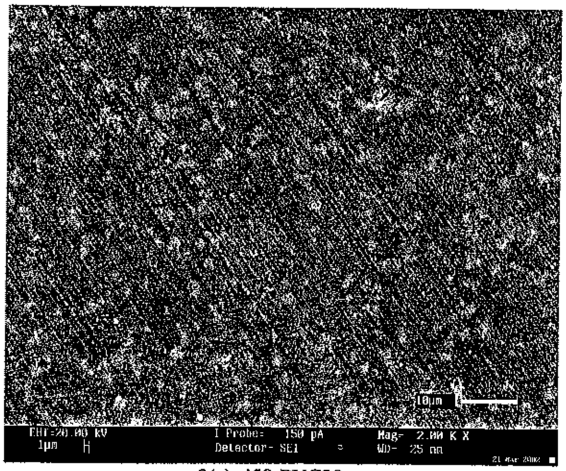


2(e) Si50-PVC10-250

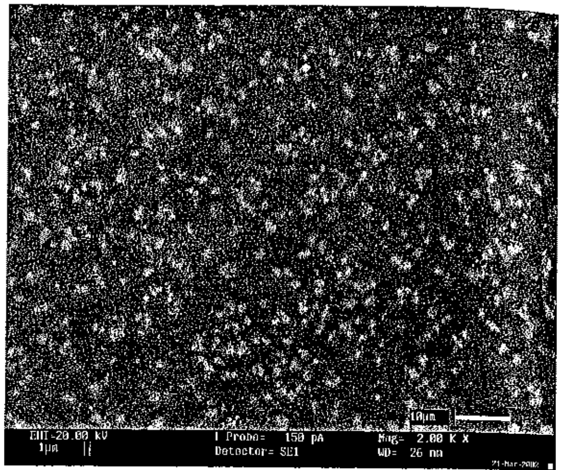


2(f) Si50-PVC10-300

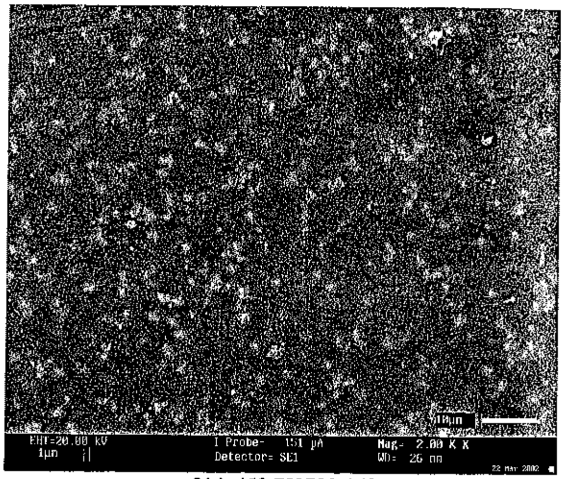
Figure 2. Micrographs of Samples with PVC10 heated at (a) Room Temperature, (b) 100 °C, (c) 150 °C, (d) 200 °C, (e) 250 °C and (f) 300 °C.



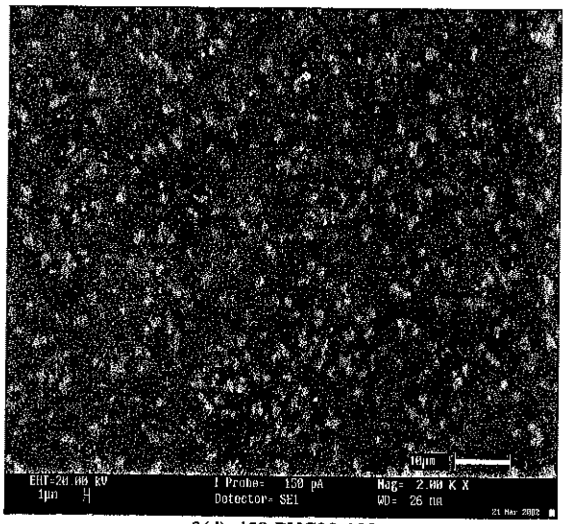
3(a) si50-PVC30-rt



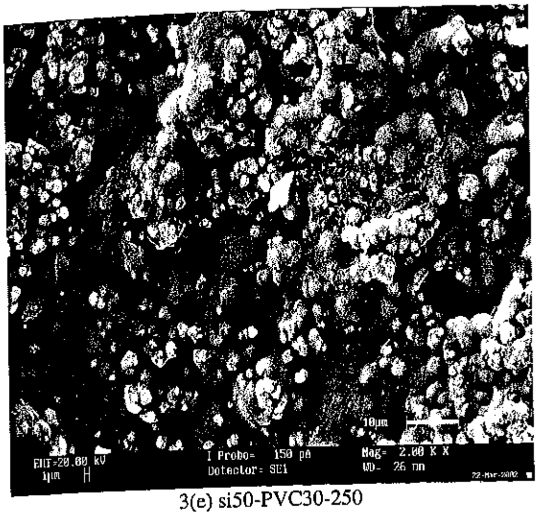
3(b) si50-PVC30-100



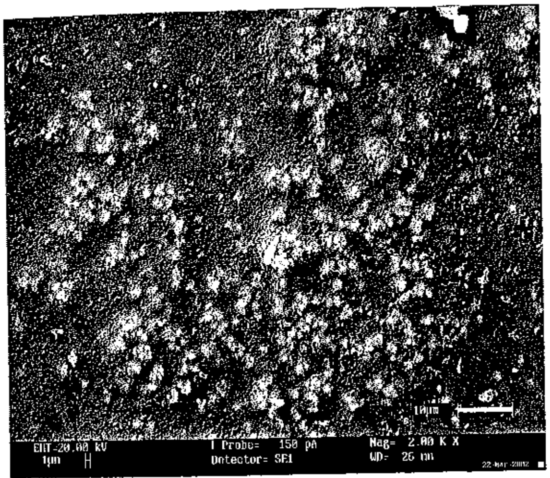
3(c) si50-PVC30-150



3(d) si50-PVC30-100



3(e) si50-PVC30-250



3(f) si50-PVC30-200

Figure 3. Micrographs of Samples with PVC30 heated at (a) Room Temperature, (b) 100 °C, (c) 150 °C, (d) 200 °C, (e) 250 °C and (f) 300 °C.

Fig. 2 and Fig.e 3 show microstructure of PVC10 and PVC30 heated from room temperature to 300°C. From the visual observation, the pigment dispersion after heating to 100°C is better than before heating. The samples after heat treatment w also showed better hardness and smoother in the film surface. This indicates that when coating film heated at 100°C, the pigment particles were re-dispersed and re-arranged along the mild steel panel surface. Further heating of the panels also helps to expose all the solvents that remained beneath the coating to form a harder and smoother coating film. This phenomenon is also applicable on the other compositions.

From the SEM micrographs, it can be shown that the pigment particles were exposed gradually from room temperature to 300°C. This phenomenon was also supported by the increase in the roughness of coating films ; surface becoming rougher, indicating a change in porosity and the presence of another corrosion product [14]. After 24 hours heating at 300°C, PVC10 (micrograph taken at magnification of 2000x) has clearly show that cracks have occurred. Scale cracking and spallation were more severe near the edges and corners than the flat surface [15]. However, there was no cracks observed at PVC30 as shown in Fig. 3.

CONCLUSION

From SEM micrograph, PVC at 30% are able to withstand weathering cycle with no chalking and yellowing. However, gloss reduces as pigment volume increases. High PVC at high silicone percentage leads to the formation of micro-cracks.

SEM results also show , cracks appearing at 300°C for PVC10. This shows that paint made from si50 with PVC30 has the best performance to weathering and heating cycle.

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