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Stress Whitening in Polyester Melamine Coatings

Role of time and temperature in industry thermosetting polymer coatings

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Stress whitening is a long-standing problem and scientific work has focused on evaluating causes of this in bulk polymer systems. In this paper we focus on this optical defect exhibited by a complex thermosetting polyester melamine coating system used extensively in the pre-coated metal industry. There are several mechanisms proposed for how stress whitening occurs and hence there is uncertainty over the causes in the systems mentioned. The most likely explanation given to date is that a number of proposed micro-mechanisms exist, which one is occurring is entirely dependent on the system being investigated. The work presented shows that the presence of dissimilar particles is the cause of the stress whitening. The proposed mechanism for whitening and its disappearance in this case is a time and temperature dependent change in density, i.e. cracking or voiding, where the cracks are outside the range that scatters light with an increase in temperature.

1. Introduction

1.1 Pre-coated Steel

Pre-coated steel, produced by coil coating, has uses within a number of industry sectors including architecture, transport and the domestic appliance (DA) market (1–3). The coil coating market in Europe uses approximately 180–200 kilotonnes of paint per annum which is worth almost €1 billion (1). This work focuses on the requirements of the DA market in which polyester melamine pre-coated systems are utilised. Pre-coated sheets are formed during the fabrication of panels into the desired shape. During this forming stage high strains will be experienced by the coating. Forming can lead to various defects including cracking, tearing and voids (4–7). In some systems a visual problem called stress whitening can arise from these defects (3).

Pre-coated steel consists of several layers. In this instance the substrate is pre-treated cold rolled steel with a two-coat polyester system on top, as seen in **Figure 1**. The basecoat provides the colour and other functional features such as corrosion protection, the top coat acts as a

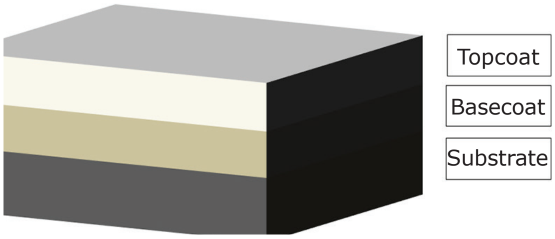

Fig. 1. Schematic diagram of a domestic appliance (DA) coating system

further barrier layer as well as adding any surface finishes required by the customer (1, 8). The basecoat is grey silver and the topcoat is a clear lacquer. Both coatings consist of a polyester resin crosslinked with hexa(methoxymethyl)melamine (HMMM), standard additives such as a catalyst and required functional additives, such as a structuring agent (1, 8). The polyester crosslinks with the melamine *via* a transetherification reaction which is thermally activated (1, 8). It is very important that the correct temperature and dwell time are used for the material, if the temperature is too low then the melamine will not crosslink enough and the melamine is also found not to distribute evenly (8). If the temperature is too high or the dwell time too long then the coating over-crosslinks, meaning it is too rigid for forming and the coating will crack, the coating often becomes brittle and has poor adhesion as well. At extremes of time or temperature, thermal degradation, shown by yellowing, occurs (1, 8, 9).

1.2 Stress Whitening

Stress whitening is the effect seen when an initially non-white polymeric material undergoes mechanical deformation, applying stress to the system, which then causes the material to exhibit a greater optical brightness and appear white, as can be seen in **Figure 2**. This effect is caused by light scattering: a variation in the reflection of light from either the surface or the bulk caused by a change in the coating (10–17). Features shown to cause stress whitening have been found to be around the range of visible light in size by Misra and co-workers (11, 18). However results within the present authors' research group generally agree more with Thiele *et al*. (19) who find scattering materials have particle sizes around 170–360 nm, which is around the range of visible light halved.

Fig. 2. Stress whitening induced in coating systems

There are several proposed changes which are attributed to stress whitening. The two categories they fall into are structural changes or changes in density (15, 16). Structural changes are seen in semi-crystalline polymers and are associated with changes in crystal orientation (15, 16). Cherry *et al*. (15) have concluded that the primary mechanism by which high density polyethylene (HDPE) stress whitens is a change in structure, associated with permanent plastic deformation. Changes in density have been seen in a plethora of polymers and include voids, crazes or cavities among others (11). Owing to the nature of the coating as a thermosetting system and therefore amorphous (8) in structure, only changes in density will be investigated in the present study.

As an example of differing density mechanisms in different polymers, Young and Lovell (14) have found stress whitening in high impact polystyrene (HIPS) and rubber toughened poly(methyl methacrylate) (RTPMMA). HIPS exhibited more whitening than RTPMMA. Scanning electron microscopy (SEM) micrographs of the two materials were compared and crazes emanating from dissimilar rubber particles in bulk polymer surface were seen in HIPS, however no crazing was visible in RTPMMA. The RTPMMA has much smaller rubber particles at 200–300 nm. The authors concluded this was the reason no crazes were emanating from the rubber. Their secondary conclusion, that cavitation within the nanoparticulate rubber was the cause of observed whitening, is in agreement with the findings of Breuer *et al.* (16), who evaluated rubber-modified polyvinyl chloride (PVC) which was shown to be stress whitening by cavitation occurring in the rubber modifiers. Contrary to the conclusions of Young and Lovell (14), the further work conducted by Breuer *et al.* (16) found that variation of the polymer was most likely to be the cause of a change in mechanism as opposed to the size of rubber particles, which the group have associated with the extent of stress whitening. Breuer *et al.* (16) also cited the crazing mechanism as the source of stress whitening in polystyrene.

Misra and co-workers (10, 11, 18, 20) have published a number of studies on stress whitening, including investigation on polyethylene and polybutylene and the effects of various additives on these polymers. Misra *et al.* (10) have stated that the primary factors influencing stress whitening are composition, molecular weight, percentage crystallinity and phase transformation. The effects seen in materials in regions of tensile strain where stress whitening is visible include, but are not limited to wedges, tearing, crazes, cracking and fibrillation. In an early paper Misra *et al.* (21) concluded that small voids will scatter more visible light and yield a whiter appearance whereas larger voids absorb in the visible light range and reduce whitening; however Misra *et al.* (11, 22) later went on to state the complexities of the relationship between light scattering and deformation and the dependence upon refractive index, surface roughness and the already mentioned void size.

There are limited publications discussing stress whitening in thermoset polymers (3, 23), and these do not explore the mechanism of whitening or contain a useful level of detail; however stress whitening has been seen as a prevalent problem within the thermoset coatings industry for many years.

1.3 Summary of Literature Findings

From the preceding review it has been found that, to date, research has investigated stress whitening in homopolymer systems (15, 24) or single modifier polymer systems (10, 12, 16, 20, 24–26). Complex systems such as coatings have not been investigated to confirm whether or not the same mechanism(s) are occurring. Tensile strain in coatings has been heavily investigated (4, 27); previous foci have been essential work of fracture (3, 28), fracture mechanics (29) and other physical properties such as viscoelastic responses (2, 30), however there has not previously been a focus on evaluating visual concerns in coatings. This work is the start of research into the visual phenomenon of stress whitening appearing in complex coatings systems, with the aim of identifying the cause of stress whitening and determining its potential mechanism(s).

1.4 Hypothesis

Industry hypothesises that stress whitening in coatings is largely due to heterogeneous particles in the bulk resin mixture. These could be mineral additions for pigmentation or strengthening, anti-corrosive pigments or a range of other additives and extenders (1). Given the mechanisms presented for various polymer systems, the heterogeneous particles hypothesis seems a reasonable assumption; however it is important to monitor both the bulk system and the additives currently included as both can affect the mechanism and occurrence of stress whitening.

In order to establish the exact cause of stress whitening in DA systems the coatings used were consecutively reconstructed by adding different component parts, such as catalysts, to the bulk resin. Functional agents were added separately, and in combination, to determine the cause of stress whitening. Once this was completed and the cause attributed, then a likely mechanism for stress whitening was established.

1.5 Strategic Choices

The coating used was a DA coating, which had been known to stress whiten under set circumstances. The ingredients in the coating were needed to achieve the specifications of the DA market. These include a polyester melamine crosslink resin system as the main polymer, solvents, a structuring agent and a liquid dispersion of particles used as a slip additive. **Table I** highlights key details of the resin mixture and the two heterogeneous additives.

Table I Topcoat Components

Name	Purpose	State	Appearance	Approximate particle size, μm	Glass transition temperature T_g, °C
Resin mixture	Binder	Viscous liquid	Transparent pale yellow	n/a	Onset 31 Midpoint 37
Additive 1	Structuring agent	Powdery solid	Opaque white	Up to 80	n/a
Additive 2	Slip additive	Dispersion of particles	Opaque white	30	n/a

2. Method

2.1 Coating Procedure

In stress whitened systems previous studies have shown that there is a correlation between defect formations from tensile strain and the presence of dissimilar particles in the polymer system. In these coatings the most likely cause of stress whitening is a particular additive. The formulae of two polyester melamine paints, which make up the two coat DA system, were analysed, and the most likely particular additives were noted.

To determine if stress whitening was linked to any particles in the systems, different combinations of the coatings were made. The focus was on the presence or absence of these particular additives in the system in order to verify which ones, if any, were causing stress whitening. All possible variations were investigated. **Table II** shows the differing combinations made for the topcoat. The full topcoat system featured a basic polyester melamine resin mixture, including solvents, catalyst and defoamer, which was homogeneous. The slip additive and structuring agent, also included, were both heterogeneous to the resin mixture.

Table II Topcoat Formulation Combinations

Name of coating	Included in coating		
	Resin mixture	Slip additive	Structuring agent
Coating 1	✓	✓	✓
Coating 2	✓	×	✓
Coating 3	✓	✓	×
Coating 4	✓	×	×

2.2 Panel Manufacture

Panels of the various coating permutations were manufactured; the substrate used was 0.6 mm thick cold rolled steel. The basecoat and topcoat were applied using a standard draw down method. The basecoat had a dry film thickness (DFT) of 13 ± 3 μm. The topcoat had a DFT of 15 ± 3 μm.

The following method of curing, for the coatings, mimics a coil line cure. For the basecoat, panels were placed in a paint oven at 300°C for 35 s and then removed and quenched with a heat sink. For the topcoat, panels were placed in a paint oven at 300°C for 40 s and then removed and quenched in water. The basecoat peak metal temperature (PMT) was 216–224°C. The PMT for the topcoat was 241–249°C.

The panels underwent basic industrial quality control tests, which showed the additions or lack of have no overall effect on the mechanical performance of the system. However, there was a difference in surface appearance. Coatings 1 and 2 containing the structuring agent had a wrinkled or textured surface, whereas Coatings 3 and 4 produced a smooth and shiny surface. Coating 1 had a more patterned wrinkled surface than Coating 2 and is reminiscent of Swiss cheese.

2.3 Induction of Stress Whitening

For this system, the method of inducing stress whitening required samples to be cooled to –20°C and stress applied. To determine the best method of applying stress, Coating 1, as the full system, underwent various tensile tests. The tests chosen were all industry standards for testing coatings (1). The most consistent test results were seen with either a cylindrical or conical mandrel bend, as seen in **Figure 3**. The cylindrical mandrel bends a sample not more than 1 mm thick and 50 mm wide through an angle up to 180° over a period of 2–3 s. Different width cylinders exist, in this case a diameter of 16 mm was used. This causes a shallow bend angle of 160° ± 3°. The conical mandrel bend consists of a 20 cm long metal cone with an initial diameter of 3.2 mm increasing up to 38 mm, the mandrel conforms to the DIN EN ISO 6860 test standard. Erichsen cup draw tests and tensile pull tests among others were also investigated; however it was not possible to consistently whiten Coating 1, ruling out the use of these methods.

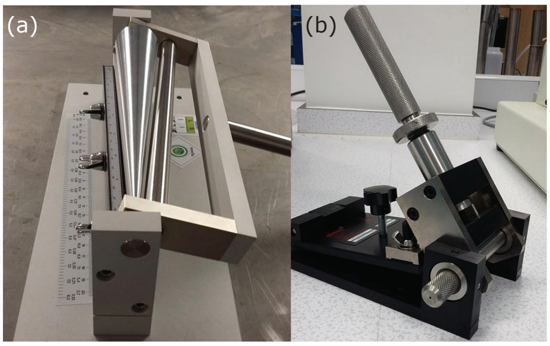

Fig. 3. (a) Conical and (b) cylindrical mandrels used to induce stress whitening

2.4 Microhardness Testing Procedure

In order to explore any influence of particle hardness, each coating underwent microhardness testing, as well as samples of the additives. Hardness testing was completed using a Fischer microhardness tester at 19 ± 0.5°C where the diamond tip forms a 2 μm indent into the surface. Once 2 μm has been reached the indenter is held in place for approximately 60 s and then removed. A probe in the indenter feeds results back to the WIN-HCU® software, which processes the hardness curves and provides a measurement of hardness. The software made nine random indents across the surface of the panel using this method, which then generated an average hardness as well as the standard error. To ensure comparability, this method was used for both the coating panels and the additives. To increase reliability, three panels of the same sample were manufactured, all of which underwent hardness tests. The results were collated and an average value determined. These results will be discussed in Section 3.2.

2.5 Scanning Electron Microscopy Procedure

SEM was used to evaluate nanofeatures on the coating surfaces. SEM imaging of samples was conducted on a Jeol 7800F FEG-SEM. The operating parameters were 2 kV accelerating voltage with the lower electron detector set with +300 V bias at an approximate working distance of 7–10 mm.

Samples were prepared from the desired coated panels by cutting a small section from the middle. The top was then cleaned using compressed air. Samples were mounted on an appropriate platen for the SEM and secured using electroless silver paint, also referred to as Silver DAG (Agar Scientific Ltd, UK). Finally, the mounted samples are sputter coated with 2–3 nm of platinum using an Agar high resolution sputter coater.

3. Results and Discussion

3.1 Stress Whitening Results

In every system that stress whitened, whitening was found to be transient and was no longer evident upon warming to room temperature or after a prolonged period at the lowered temperature. Re-cooling the samples did not make whitening re-appear.

The topcoat combinations were tested. None of the coatings stress whitened at room temperature. Both Coatings 1 and 2 (**Table II**) exhibited stress whitening after being cooled to –20°C for at least 1 h, and bent, as seen in **Figure 4**. Whitening did not last for more than a minute when in ambient conditions and did not last for more than one month when immediately re-stored at –20°C. A conical mandrel and a cylindrical mandrel both induced stress whitening in the samples. The stress whitening appeared speckled in both Coatings 1 and 2. For Coating 1 specifically the white speckles appeared to be at the base of the craters or 'Swiss cheese' like holes. A single additive was shown to be responsible for stress whitening, this was additive one or the structuring agent.

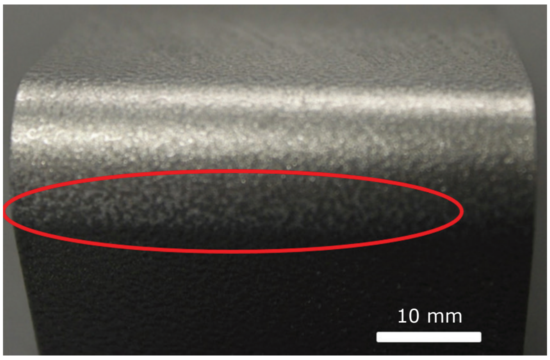

Fig. 4. Transient stress whitening caused by structuring agent

Mandrel testing revealed that the basecoat did not stress whiten for any combination, it was ruled that only the topcoat stress whitened. However the silver pigment in the basecoat may have enhanced the appearance of stress whitening in the topcoat.

Characterisation of the coatings was conducted to test for property variation and to highlight any differences between the additive and the coating. The only consistent element between Coatings 1 and 2 that was not present in Coatings 3 and 4 was the structuring agent, producing the conclusion that the presence of the structuring agent causes stress whitening.

3.2 Microhardness Testing Results

The hardness results listed in **Table III** show no statistically significant variation between Coatings 1 through to 4; however the additives were significantly softer than the coatings. Given that the structuring agent was found to be the source of stress whitening, it is notable that the coating was significantly harder than the structuring agent. Variation in hardness can produce different reactions to strain, where a hard system has a short elastic response followed by a viscoelastic and plastic response; a soft system is more likely to have a greater elastic response, with only a small amount of viscoelastic or plastic response occurring. The presence of heterogeneous particles has been cited as the initiation point for various defects.

Table III Hardness Measurements Accrued from Micro-Hardness Tester

Sample	Marten's Hardness, N mm^{-2}	Standard deviation, N mm^{-2}
Coating 1	170	22
Coating 2	175	25
Coating 3	164	14
Coating 4	176	5
Slip additive	19	3
Structuring agent	24	9

3.3 Scanning Electron Microscopy Analysis

SEM images have been captured for all of the samples. These were before bending or tensile testing had occurred. The SEM images were used as a comparison to isolate whether any particles can be seen that correlate to the structuring agent. SEM was also used as a stage of elimination, as any particles or surface artefacts only present in Coatings 3 or 4 were not causing stress whitening and so can be ruled out in the other systems. The 20–70 μm circular craters visible in Coatings 1 and 3 are an example of an artefact not associated with the structuring agent, meaning they cannot be the cause of whitening. The circular craters in fact correspond in size with either a single particle or an agglomeration of the slip additive and are most likely caused by this. **Figures 5** to **8** show overview SEM micrographs of Coatings 1 through 4.

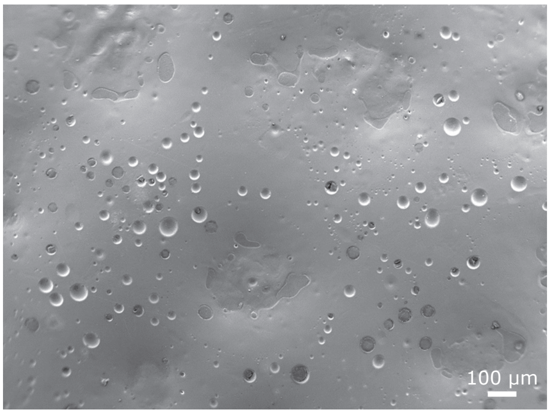

Fig. 5. SEM micrograph of Coating 1 before bending. Coating 1 is the full system and the micrograph features surface artefacts associated with both slip additive and structuring agent. It also has features which do not appear in any of the other coatings, these are the amorphous shapes which are around 100 μm

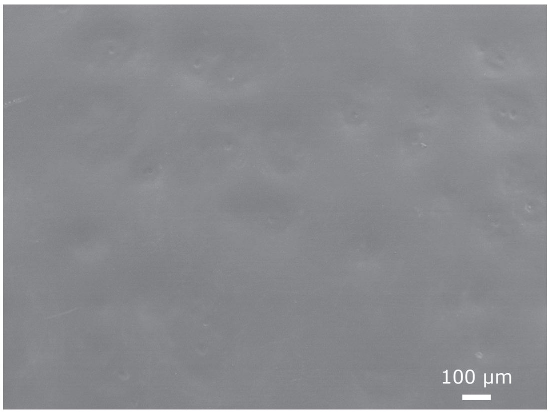

Fig. 6. SEM micrograph of Coating 2 before bending. Coating 2 only contains resin mixture and structuring agent. The circular depressions with smaller approximately 30 μm circular artefacts within them are associated with the structuring agent

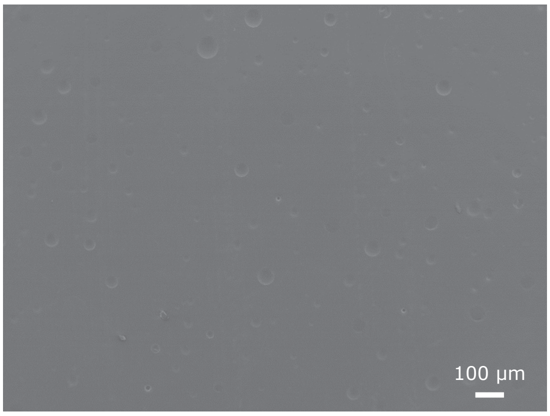

Fig. 7. SEM micrograph of Coating 3 before bending. Coating 3 contains only resin mixture and slip additive. The circular concave artefacts are attributed to either single particles or agglomerates of the slip additive. They have a size range of approximately 20–70 μm

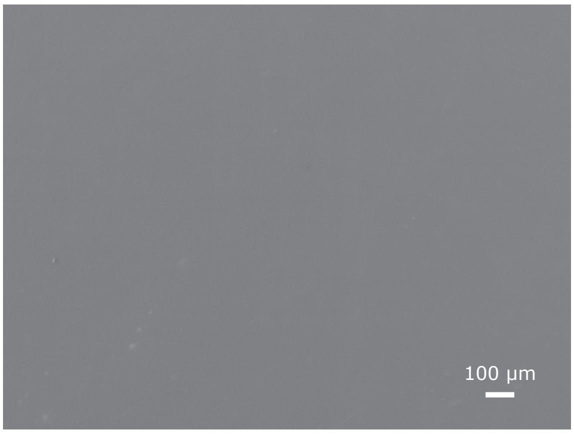

Fig. 8. SEM micrograph of Coating 4 before bending. Very few if any surface artefacts are visible. There are no repeating artefacts across the entire sample, leading to the conclusion that any individual artefacts are either dirt or specific surface defects into the coating

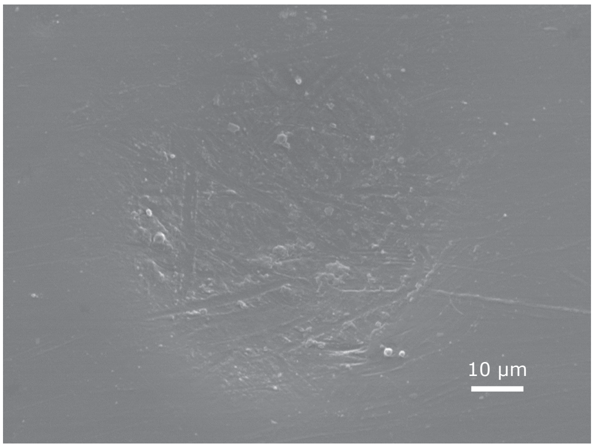

Fig. 9. SEM micrograph of Coating 2 before bending. This shows a close-up of the approximately 30 μm surface artefact, which is potentially structuring agent

Figure 9 shows a surface artefact only found in Coatings 1 and 2. This is the most likely artefact that can be attributed to the structuring agent, which is the cause of stress whitening. These artefacts will be the point of focus in SEM studies of the bent coatings.

Figure 10 is a schematic representation of the proposed mechanism for stress whitening in this heterogeneous polyester melamine coating. Given the transient nature of the stress whitening, a feasible hypothesis is that the particles of structuring agent and the polymer are responding differently to the stress on the system. The different responses are time dependent, i.e. kinetic, meaning the initial response is not the material's final response and after a short time the material reaches equilibrium. At this equilibrium the conditions for stress whitening are no longer present, unlike in the initial response, meaning stress whitening is no longer visible. This would also account for the lack of stress whitening at a warmer temperature, as the system will go through elastic and visco-elastic changes far more quickly than when at –20°C.

The most likely scenarios are either a self-healing effect where any nano-cracks or defects present in the correct range are sealed by the final time-dependant viscoelastic response of the resin matrix and particle to the strain applied; or the nano-cracks or defects in the coating have become larger than the range which interferes with light

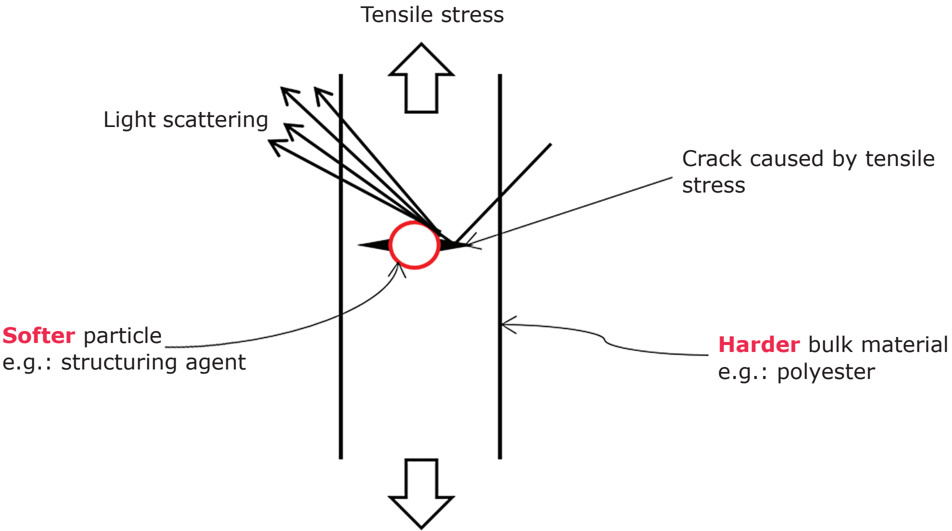

Fig. 10. Proposed mechanism of stress whitening in heterogeneous coating system

reflection as equilibrium is reached in cases where the two materials have different end reactions to strain. Due to the transient nature of the stress whitening, determining which is true for this system is challenging.

4. Conclusions

In conclusion, the structuring agent present in the coating caused stress whitening when tensile strain occurred in the system. The structuring agent is a softer particle, which is not detectable by microhardness in the bulk coating system. Previously industry has only observed stress whitening in systems with harder heterogeneous particles. Stress whitening is caused by light scattering off defects present in the coating; the most likely defects are voids or nano-cracks around the heterogeneous structuring agent particles, which act as an initiation point for defects. The transient nature of the stress whitening is most likely attributed to this difference in hardness. SEM micrographs show surface features indicative of a potential point of defect, surface comparison has ruled out many surface artefacts.

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Dr Chris Lowe moved from photo-resists to drugs for African cows and then to paints that cure under the influence of UV light. These early experiences have informed his research interests into areas such as structure property relationships for both weathering resistance and mechanical properties, adhesion and the lack of it and the application of spectroscopic tools in general to issues with paint.

Professor David T. Gethin DSc, CEng, FIechE, FLSW, undertakes fundamental and applied research in all aspects of printing and coating processes with a key interest in the deposition of functional materials. This includes the formulation of inks, their deposition using the appropriate processes and metrology of the printed layers or devices. This work covers a range of technology readiness from laboratory scale exploration to scale up for industrial application.