



The influence of basecoat pigmentation on chemical structure and surface topology of automotive clearcoats during weathering

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ABSTRACT

The present study aims at elucidating the effect of basecoat pigmentation on the chemical structure and surface topology of its attached clearcoat during weathering exposure. Two extremes of differently behaved basecoat pigmentations (i.e. silver and black) were chosen. Different analyses such as FTIR, ATR, surface energy measurements and AFM were carried out on such coatings after they were subjected to accelerated weathering conditions. It was found that the black basecoat procured more post-curing reactions to the attached clearcoat at initial weathering times, while the silver basecoat induced higher degrees of photodegradations during the whole time of weathering. Such inductions were attributed to the inherent absorptive or reflective behavior of the black or the silver basecoats toward incident radiation.

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1. Introduction

Excellent appearance and aesthetic properties are of utmost importance in the automotive industry, since these seem to have a direct bearing on the appeal and purchases of automobiles. Enhanced aesthetic properties have been achieved by introducing the basecoat/clearcoat (BC/CC) automotive finishing systems which reduce mottling and orange peel defects [1–3]. However, it should be mentioned that retention of such aesthetic properties during service life is as important as its initial creation.

It is well known that mechanical and/or environmental factors could spoil the appearance of automotive coatings, especially the clearcoats. Environmental damages are caused by various combined exposures to sunlight, temperature variations, humidity, biological substances and acid rain which could occur for several hours during day and/or night [4]. In recent 20 years, there have been lots of attempts to study environmental degradation of automotive coating systems in both natural exterior and simulated conditions [5,6]. These attempts resulted in outstanding findings about degradation mechanisms, development of new materials to protect the coating and novel models to predict the service life

of the automotive coatings [7–9]. It was revealed that sunlight and humidity induce various chemical reactions such as photodegradation and/or hydrolytic reactions within the coating. The mechanisms of photo and hydrolytic degradation as well as their impacts on mechanical [10–15], physical [16] and electrochemical properties [17] of the coating system have been extensively studied [18–21].

Due to significant role of clearcoat, most previous studies have focused only on clearcoat layer, not investigating underneath layers or their role on degradation of clearcoat [22]. There are few papers studying the degradation of other layers, i.e. basecoat, primer or their influence on clearcoat degradation [23,24]. However, there is reason to believe that the basecoat could greatly affect the weathering performance of its attached clearcoat. In order to illustrate how a basecoat could vary the weathering performance of a clearcoat; it is necessary to clarify how various basecoats would react to incident radiation. A common basecoat would contain colored pigments and/or metallic flake. Colored pigments absorb and/or scatter incident visible radiation reaching the bulk of the basecoat, according to their color, size and refractive index. Metallic flakes, in accord to their level of orientation, reflect and/or scatter incident radiation only at the surface of the basecoat. In this manner, fractions of returned incident light passing through the clearcoat are decisive in causing chemical structural changes in the clearcoat.

In the present study, attempts were made to elucidate the influence of basecoat pigmentation on variations in chemical structure and surface topology of a typical automotive clearcoat during

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weathering. To this end, two extreme acrylic/melamine basecoats separately having a silver or a black pigmentation were selected. A silver basecoat is characterized by presence of high loads of aluminum flakes and absence of colored pigments, in which the chance of reflecting incident light is high (i.e. high reflectivity) and the chance of absorbing incident light is minimal (i.e. low absorptivity). While the black basecoat, is characterized by the presence of high loads of a black pigment (i.e. high absorptivity), and a lower load of aluminum flakes (i.e. lower reflectivity). Based on this viewpoint, other basecoats depending on their ability to reflect or absorb incident light would be rated to lie somewhere between the chosen black and silver extreme basecoats.

Several previous works have demonstrated that clearcoat degradation in the bulk of the clearcoat occurs to a lesser extent than its surface [11,25,26]. Therefore, variations in chemical structure at the surface as well as the bulk of the clearcoat after various weathering exposure time intervals were also assessed using a standard in a predetermined accelerated automotive weathering test.

2. Experimental

2.1. Investigated paint systems

In order to determine bulk and surface characteristics of clearcoats, two series of samples were prepared. The first series were prepared according to a complete automotive coating system applied on pretreated steel panels and the second series were free film samples applied on glass panels. For the complete system, steel panels were phosphated and were then coated by an epoxy amine electrodeposited (ED) layer. Afterwards, a poly-ester/melamine primer was applied over ED layer. The thicknesses of ED and primer layers were about 20 and 30–40 μm , respectively. As was mentioned previously, two acrylic melamine basecoats (i.e. silver and black) were chosen and applied on the primer. After a short flash time, an identical acrylic melamine clearcoat was applied on top of the basecoats by the wet on wet method. In this method, clearcoat layer is applied over uncured basecoat. After a short time for solvent evaporation and when the basecoat is surface-dried, the clearcoat is applied over basecoat and their curing process is performed simultaneously. The simultaneous curing process for the basecoat/clearcoat system was carried out at 140 °C for 20 min. The thicknesses of the cured base and clear coats were 12–15 and 40–45 μm , respectively. For the free film samples, only the last two layers (i.e. basecoat and clearcoat) were applied on glass panels and cured accordingly. After curing, the free films of base/clearcoat were obtained by removing them from the glass panels. The samples were coded by XS and XB referring to whether the clearcoat was applied on a silver or a black basecoat, respectively.

2.2. Weathering conditions

An Atlas Xenotest Beta LM weather-o-meter was used, to simulate outdoor exposure. It employs a Xenon arc light source with inner and outer quartz/borosilicate filters. Weathering conditions in accordance to the Peugeot D27 1389-95 standard, were exerted. The weathering cycle comprised of continuous irradiance of 0.55 W/m^2 at 340 nm at an interval of 108 min of dry condition (relative humidity 50%, dry temperature 54 °C) which was followed by 18 min of deionized water spraying on surface of specimens. After various exposure times (0, 150, 300, 450, 600 and 1000 h) the samples were removed and tested.

2.3. Tests

FTIR spectroscopy was used in order to analyze chemical variations caused by weathering at various exposure times in the bulk

of clearcoats coated on silver and black basecoats. For this purpose, samples were prepared by scraping away the surface of the weathered samples by a surgery knife, reaching to bulk of the coating. In our previous work [11], it was demonstrated that the degradation in clearcoat can be divided into 3 sections; surface, bulk and clearcoat/basecoat interface. It was confirmed that variations within each of these sections are not significant. Based on this finding, sample preparation was precisely performed so that to obtain the sample from neither the surface nor the base/clearcoat interface. The scraped powders were subsequently mixed with KBr to obtain pellets for FTIR analyses.

Also, in order to follow variations in chemical structure of the clearcoats' surfaces, attenuated total reflectance (ATR) spectroscopy was used. The respective attached basecoat/clearcoat free films were used in ATR spectroscopy. Both FTIR and ATR spectroscopies were carried out on a Bruker Spectrometer model IFS48 instrument. The internal reflection element used in ATR instrument was a rectangular ZnSe crystal.

Surface energies of clearcoats were measured by the aid of a Kruss G40 contact angle measuring system. Surface energies were calculated according to the Wu equation; using water, formamide and diiodomethane as probe liquids (their characteristics are well known). Surface topology of weathered clearcoats, was studied by a DME Scanner AFM microscope model DS 95-50.

3. Results and discussion

3.1. Chemical analyses

3.1.1. Coatings' bulk

Chemical changes in the bulk of two identical clearcoats coated separately on silver or black basecoats, after various exposures to artificial weathering, were carried out by FTIR analysis. Fig. 1 illustrates such changes.

Comparative changes in NH&OH (around 3000–3600 cm^{-1}), carbonyl (1600–1700 cm^{-1}) and etheric bands (1000–1200 cm^{-1}) are of considerable importance for deduction purposes. In order to make this comparison possible, the intensity of above-mentioned peaks were normalized with respect to the intensity of an unaffected peak. By taking the CH vibration peak around 2800–3000 cm^{-1} as an internal reference, the under peak area of NH&OH, carbonyl and etheric bands in each sample were divided by the under peak area of its own corresponding unchanged CH peak. Fig. 2 shows such normalized absorbances of etheric, carbonyl and NH&OH bands of clearcoats attached to silver or black basecoats.

The relative initial normalized absorbances of clearcoats coated on silver or black basecoats before weathering (i.e. exposure time = 0) clearly indicate an inherent differences in the bulk of these clearcoats. The normalized etheric absorbances for the clearcoats attached to silver and black basecoats are 0.74 and 0.67 respectively. It clearly reveals that a clearcoat attached to a silver basecoat has a larger etheric (and smaller NH&OH) absorption bands than the one attached to a black basecoat. This larger etheric absorption as well as the correspondingly higher thermo-mechanical characteristics of the clearcoat attached to a silver basecoat can obviously reveal a higher degree of cure of this clearcoat. These chemical evidences together with higher hardness and cross-link density values [10] for clearcoat having a silver basecoat compared to that having a black basecoat confirm the more efficient curing process on a silver basecoat. All these evidences reveal that there is such difference between clearcoats which are cured on different basecoats. This leads to the belief that type of pigmentation present in the basecoat may influence heat transfer induced by conduction, convection or radiation of the clearcoats' curing process. The heat needed for the curing of the coatings was supplied by two different

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