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Surface analysis of matt powder coatings

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Abstract

A study of gloss reduction of powder coatings was performed using different waxes, fillers and non-compatible polymer resins (matting hardeners). Coatings with reduced surface smoothness were analysed. Scanning electron micrographs were taken to visualize the surfaces. All of the applied matting additives changed the surface profile of coating. The most interesting surface structural form is provided by wax particles. The average size of these forms was calculated by applying image analysis to some sample micrographs. The surface shape of all of the prepared coatings was analysed using surface profile measurements. Two parameters of these profiles were determined, the average roughness and the mean spacing between the surface peaks. The influence of several physical surface parameters on specular gloss characteristics is discussed.

Keywords: Powder coatings; Matting; Specular gloss; Gloss reduction; Surface analysis

1. Introduction

No measuring instrument is known to be capable of measuring the details of the appearance of a surface as seen by the human eye. Therefore, various methods have been proposed to represent the appearance of a coating and of a coated object [1,2]. The visual impression of a surface is recognized as various types of glossiness that are frequently classified as different degrees of gloss. Several criteria are used in glossiness rankings. Among these, in coatings the specular gloss (mirror-like) is the most often considered [3,4]. The specular gloss measures the intensity of mirror-type reflection, i.e. when the angle of incidence is equal to the angle of reflection. The more the light is reflected this way, the greater is the impression of gloss. Smooth and polished surfaces reflect a major part of the light in this way. On rough surfaces, the light is scattered to several directions. Therefore, the image forming quality is diminished and the reflected image is blurred.

Low-gloss coatings are popular when used for aesthetic and functional purposes. A surface with lower gloss hides surface imperfections in a way that is superior to the hiding provided by a surface of higher gloss. The application of a low-gloss coating might, therefore, be easier to achieve than that of a glossy one and be more cost effective. This is one of the reasons why matt finishes are produced.

Several techniques are known to produce coatings with lower gloss finishes. They can be divided into two basic categories, whether they act due to the presence of non-soluble particles or whether they are effective due to the addition of resinous materials that demonstrate different compatibility behaviour. Gloss reduction using non-soluble particles is widely used in liquid-based coatings. During drying processes, the loss of solvents and condensation products results in a fairly high shrinkage of the coating film. Consequently, a diffusely reflecting micro-textured surface is formed [5–8]. This matting technique is less effective in coatings that undergo little shrinkage during film formation. Such coatings include powder coatings (PC) and some types of UV curing systems where the production of matt and low-gloss finishes is not straightforward. Here, resinous materials are frequently applied that

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shows limited compatibility with the binder. This causes the formation of a coating that has an imperfectly smooth surface [9–11]. In addition, fillers with larger particle sizes are frequently applied to reduce the surface smoothness.

One of the more important properties of PC is that there is little or no volatile organic compounds (VOC) emission. Thus, PC products are considered to provide the most environmentally friendly coating system. In the past, a method for evaluating the differences in the degree of dispersion in PC has been proposed [12–14]. The method gives the average particle size and the particle size distribution. Also studied has been the influence of the input power of the extrusion equipment on the state of the pigment dispersion in PC [14]. Quantitative information was obtained using selective oxygen plasma and by evaluating the scanning electron micrograph (SEM) by image analysis [15–18]. Recently, the bonding-process efficiency and Al-flake orientation, during the curing of PC, were studied [19]. The orientation of flakes was evaluated during melting of the coating powder and was accomplished already at 100 °C. All these studies were performed on samples from a standard PC production line.

This current study was performed on the samples from standard production process in order to evaluate the various types of matting phenomena and to establish the role played by the materials used in the production of PC.

2. Experimental

2.1. Samples

The carboxy-polyester binder was used in all PC samples. Different matting additives were applied: micronised waxes, fillers and polymer resin (matting hardener). The data provided by the manufacturers are summarised in Table 1. The application of these matting additives in coating powder samples is given in Table 2. Approximately the same net concentration of matting additives was applied in all samples (9 vol%). The coating powders were prepared on a standard PC industrial production line applying the twin-screw extruder ZSK 50 with 43.2 kW of input power.

The powder samples were sprayed electrostatically onto aluminium sheets. They were cured for 10 min at 180 °C. The thickness of cured coatings was 60–70 μm. In addition, selected powder samples were sprayed over the aluminium substrate (50 cm × 50 cm) of the same thickness and cured in a gradient bar oven for 10 min. The temperature gradient was adjusted to vary nearly linearly from 80 °C at one end of the sample up to 180 °C at the other. This way, test panels with curing temperatures in the range from 80 to 180 °C were prepared.

Some samples were selectively etched to remove the topmost layer of the binder from the coating surface leaving all the other particles unchanged. The procedure was based on selective interaction of gaseous particles with coating film in a weakly ionised highly dissociated oxygen plasma [15,16].

Table 1
Description of applied matting additives (data from the producers)

Matting additive	Chemical nature	Particle size	Softening point (°C)
Wax A	Ethylene homopolymer wax		104–108
Wax B	Micronized polyolefin wax		109–117
Wax C	Micronized modified polyethylene wax	$\overline{d}$ = 8 μm, <35 μm	135
Wax D	Micronized polypropylene		154–157
Wax E	Micronized poliolefin wax		
Filler 1	$CaCO_3$	$\overline{d}$ = 5–6 μm, <20 μm	–
Filler 2	Complex mineral, consisting of mica, chlorite-containing slate and quartz	12 μm	–
Filler 3	Micronized aluminium hydroxide, $Al(OH)_3$		–
Polymer resin (hardener)	Mono-salt of a polycarboxylic acid and a cyclic amidine		210–235

All materials were used in powder form. Not all the data are available.

2.2. Measurements and calculations

Three surface properties were measured, the specular gloss, the surface profile and the topography of the coating surface.

The specular gloss is given by the ratio of the two fluxes reflected in the selected specular direction, one on the sample and the other on the standard (polished black glass with a refractive index of 1.567). The readings are expressed in GU (gloss units). The calibration was performed on the black gloss standard. A Micro-TRI-Gloss portable glossmeter measuring unit (BYK-Gardner) was used in this part of the study. All of the measurements were carried out using the 60° incidence angle.

The surface profile of the coating samples was measured using a profilometer Talysurf (Rank Taylor Hobson Series

Table 2
Matting additives used in formulations of PC samples and measurement results: the gloss, the average diameter of wax particles on a surface of a cured coating (D_{wax}), the average roughness (R_a), and the mean spacing between surface profile peaks (S)

Sample	Matting additives	Gloss (GU)	D_{wax} (μm)	R_a (μm)	S (μm)
S1	Wax A	78	2.59	0.090 ± 0.011	8.77 ± 0.54
S2	Wax B	55	9.18	0.258 ± 0.028	11.39 ± 1.00
S3	Wax A, filler 1	68	–	0.126 ± 0.004	9.63 ± 0.86
S4	Wax A, filler 1, filler 2	57	3.52	0.186 ± 0.005	11.26 ± 0.25
S5	Hardener	45	–	0.242 ± 0.031	17.8 ± 1.8
S6	Wax A, wax B	72	3.31	0.111 ± 0.015	8.75 ± 0.49
S7	Wax A, filler 2	63	5.29	0.148 ± 0.004	12.10 ± 0.35
S8	Wax C, filler 1, filler 3	66	–	0.103 ± 0.002	9.05 ± 0.88
S9	Wax D, filler 3	40	14.45	0.46 ± 0.14	19.4 ± 2.3
S10	Wax E, filler 1	56	6.36	0.234 ± 0.019	12.29 ± 0.59
S11	Wax A, wax B, filler 2	74	2.95	0.144 ± 0.022	10.76 ± 0.83

Determination of D_{wax} was not possible for samples S3 and S8.