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Roughness of pigment coatings and its influence on gloss

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ABSTRACT

A robust method is used for analyzing roughness at a wide range of lateral length scales. The method is based on two-point correlation where both the amplitude and lateral spacing of surface heights are considered when determining the roughness. Atomic force microcopy and confocal optical microscopy images were captured for a set of pigment-coated samples. The effects of sampling interval, image size and filtering on surface roughness were studied. Isotropy and periodicity of roughness were determined by analyzing the angular distribution of the correlation length (*T*) and the autocorrelation function (ACF). A clear dependence of root mean square (RMS) roughness (σ) on *T* was established for randomly distributed surfaces. By taking into account the σ -*T* dependence it was possible to obtain σ for various length scales for each sample and thus attaining the most relevant σ for a certain surface function, which in this study was specular reflection of light (gloss). The roughness analysis showed that a small amount of DPP coating was sufficient to completely cover and change the surface of the substrate, while kaolin coatings gave a different response.

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1. Introduction

The quality and functionality of materials in technological applications are generally governed by three aspects: chemical composition, morphology (bulk structure; form, size and shape) and surface topography (spatial relation of surface features). Although these factors may be regarded as being of equal importance, the technological performance of materials in, e.g. heat transfer between solid bodies, optics, electrical transport, tribology (wear and lubrication), adhesion, wetting and biocompatibility is often highly dependent on surface topography and roughness [1–5].

Surfaces can be classified by the distribution and placement of topographical features. Surfaces encountered in technological applications are generally composed of roughness on various length scales, from millimeters down to nanometers, which are superimposed on each other [2,5–7]. Roughness is an intrinsic property of a surface. However, measured (effective) roughness is scale-dependent; it depends on the available measurement scale and the sampling interval of the measurement technique. This makes measured roughness essentially an extrinsic property.

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Surface roughness may be quantified by analyzing the different moments of height distribution (e.g. mean height, standard deviation of surface heights, skewness, kurtosis) [2,7–10]. However, a single roughness value is only relevant for a certain length scale, which is related to the bandwidth covered by the nominal impact width of the physical phenomenon studied [5]. Thus, roughness values obtained using single point correlation are generally of limited value in describing a surface, since they give no indication of the lateral distribution of surface heights.

The distribution of surface features can exhibit a dominant direction (anisotropy) or be completely isotropic. Furthermore, the placement of features can show periodicity or be completely random. The topography of a surface (whether isotropic or anisotropic, and random or periodic) may be defined by twopoint correlations, i.e. using both the amplitude of waveform (or roughness height distribution) and the lateral spacing (or wavelength). The parameter often used to describe the former is the root mean square (RMS) roughness (σ) which gives the standard deviation of surface heights [2]. For describing the latter, autocorrelation function (ACF) (or auto-covariance function in the case when the mean values of the height profiles are equal to zero) is often used. ACF provides two-point correlated information about the spatial relation and dependence of data and can be used to indicate randomness/periodicity and isotropy/anisotropy of surface features [2,5,11,12].





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A current topic related to coating industry is specular reflection (i.e. gloss) and its dependence on factors such as coating color formulation, particle packing, pore structure and especially refractive index and surface roughness [8–10,12–16]. It has been shown that increasing roughness (amplitude) increases the spreading of reflected light, thus increasing diffuse scattering and reducing gloss [8–10,12,15,16]. Also the lateral distribution of topographical heights (wavelength) has been shown to affect light scattering and gloss [12,16]. Problems in correlating gloss and surface roughness have been encountered related to accuracy of characterizing surface roughness (e.g. measurement techniques with insufficient resolution), as well as lack of advanced roughness analysis [8–10,15].

The purpose of the present study is to examine the effects of sampling interval, image size, and filtering on surface roughness of pigment coatings at different length scales. Furthermore, a relation between the vertical (amplitude) and lateral (wavelength) characteristics of roughness was studied for a wide range of wavelengths. In order to do this, combined topographical characterization by atomic force microcopy (AFM) and confocal optical microscopy (COM) was carried out. Finally, the roughness characteristics of the pigment coatings were correlated to their optical behavior (gloss).

2. Materials and methods

2.1. Sample preparation

A "Synthetic paper," Yupo-film (74 g/m², Mitsubishi-Yuka, Paper, Oji-Paper, Yupo Corporation, Japan), was used as a coating substrate. The film consists of three extruded polypropylene layers with inorganic filler. The middle layer has been stretched in machine direction and both outer layers have been stretched in cross direction resulting in a high surface roughness for a polymer film. The Yupo film was coated with spherical Dow Plastic Pigment (DPP) 3720 (Dow Europe Gmbh, Switzerland) with a median particle size of 260 nm given by the supplier, and platy kaolin with a median particle size of 600 nm given by the supplier (Astra Plate 100, Imerys Minerals Ltd., England). Styrene butadiene (LATEXIA 319, Latexia SB Oy, Finland) was used as binder (4 pph). The coating amounts (g/m^2) of kaolin A-E were 1.6, 2.2, 6.0, 17, 28, and for DPP 2.0. The pigment coatings were applied on the Yupo film substrate with a laboratory rod-coater (K-Control Coater Model 202, RK Print-Coat Instruments Ltd., UK), a more detailed description can be found in ref. [8]. Mica (muscovite) was used as a "perfectly" smooth reference sample for the specular reflection measurements.

2.2. Gloss measurements

Gloss was measured using a ZLR 1050 Laboratory Glossmeter (Zehntner GmbH Testing Instruments, Switzerland). Glossmeter measures the intensity of specular reflection of the sample (I_{sample}) relative to that of a smooth reference standard ($I_{reference}$) at a specified incident angle. The average gloss (G_a) is defined by the expression:

$$G_{\rm a} = 100 \times \frac{I_{\rm sample}}{I_{\rm reference}} \tag{1}$$

were *I* is the ratio of the reflected intensity I_r , and the incident intensity I_i observed by the detector [14]. A black polished glass standard with a refractive index of 1.57 was used as a reference. The studied materials have a refractive index within 1.49–1.59, Yupo-film (polypropylene) has the lowest value (1.49) and DPP 3720 the highest (1.59) [17–19]. The reported average gloss (G_a) is a mean value of 12 measurements measured at an angle of incidence of 75°

(normal to surface) with a sampling area of $14 \text{ mm} \times 6 \text{ mm}$ taken from randomly selected spots in four directions (0°, 90°, 180° and 270°) from the coating application direction. The standard deviation of the gloss measurements was ≤ 2 units. Gloss was also tested at angles of incident light of 20° and 60°. However, the diffuse reflection was too high for the rougher samples to get accurate values and differences, limiting significantly the studied gloss range. Therefore the results for 20° and 60° are not reported here.

2.3. Atomic force microscopy (AFM) measurements

The AFM measurements were carried out at ambient conditions with a Nanoscope IIIa (Digital Instruments Veeco Meterology Group, Santa Barbara, CA) atomic force microscope. The microscope was placed on an active isolation table (MOD-1, JAS Scientific Instruments), which was placed on a massive stone table to eliminate external vibrational noise. All images were recorded with a J-scanner and using silicon cantilevers (NSC15/NoAl, MikroMasch TM) in intermittent mode. The free vibration amplitude of the cantilever was set to 75 nm \pm 5 nm (high tapping), and a damping ratio (tapping amplitude/free amplitude) within 0.50–0.75 was used for the imaging. Height images were recorded using a scan speed between 0.2–1.0 Hz, with a pixel resolution of 512 \times 512. The images were captured at random spots of a large sample sheet. Images with minimal defects and asperities were chosen for further analyses.

2.4. Confocal optical microscopy (COM) measurements

The COM measurements were carried out at ambient conditions with a NanoFocus μ Surf 3D confocal white light microscope (NanoFocus AG, Germany). The images were captured using a lens with 50× magnification giving a measurement field of 320 μ m × 309 μ m with a pixel resolution of 512 × 512. The images were captured at random spots of a large sample sheet. Images with minimal defects and asperities were chosen for further analyses.

2.5. Image analysis

AFM and COM topographic images were processed and analyzed with SPIP (Scanning Probe Image Processor, Image Metrology, Denmark) image analysis software [20]. Following image sizes were analyzed: $3 \mu m \times 3 \mu m$, $10 \mu m \times 10 \mu m$ and $50 \mu m \times 50 \mu m$ (obtained by AFM) and $320 \mu m \times 309 \mu m$ (obtained by COM). The corresponding lateral sampling intervals (τ) were 5.86, 19.5, 97.7 and 625 nm, respectively. The smallest image size was chosen based on the fact that maximum effective lateral resolution for the AFM tips used here (radius of curvature ~10 nm) is around 7 nm (Rayleigh's criterion) [21], and thus a smaller image size would not yield any additional topographic information at the lateral direction.

2.6. Roughness parameters and autocorrelation function

Roughness parameters used in this study are described below [2,20]. The roughness parameters are valid for an $M \times N$ rectangular sampling area with the lateral directions x and y and vertical direction z.

The RMS deviation of surface topography (σ) is defined as

$$\sigma = \sqrt{\frac{1}{MN} \sum_{k=0}^{M-1} \sum_{l=0}^{N-1} [z(x_k, y_l) - \mu]^2}$$
(2)

were μ is the mean height:

$$\mu = \frac{1}{MN} \sum_{k=0}^{M-1} \sum_{l=0}^{N-1} z(x_k, y_l).$$
(3)

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