CHARACTERIZING THE APPEARANCE AND ITS RELATED MICROSTRUCTURE OF METALLIC-FLAKED COATINGS

by

Li-Piin Sung, Maria E. Nadal
and Mary E. McKnight
National Institute of Standards and Technology
Gaithersburg, MD 20899 USA

Reprinted from the 3rd International Conference on the Coatings for Plastics Symposium, June 5-7, 2000, Troy, MI. Proceedings. Paint & Coatings Industry Magazine, Business News Publishing Company, Troy, MI, 2000.

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Abstract

The National Institute of Standards and Technology (NIST) is conducting a study to advance the science of measuring and modeling the optical scattering properties of materials. Results of measurements and mathematical models for quantifying light scattering from a material are being used as input for computer rendering models and procedures for determining the appearance characteristics. This study will advance the characterization of spectral, goniophotometric and textural properties and improve the understanding of the interactions between light and the microstructural properties of materials. The measurement techniques and the models will be generally applicable in ecommerce and in the design, manufacture and sale of materials. This paper will briefly discuss preliminary results of a systematic study on metallic pigmented coatings using laser scanning confocal microscopy and optical scattering.

Introduction

The appearance of a material is the result of a complex interaction of the light incident on the object, the optical scattering characteristics of the material, and human perception. The optical scattering properties of the material depend on its surface topography and subsurface microstructure. Thus, identification and characterization of the microstructure of the coated materials are crucial for relating the microstructure to the optical scattering properties and for predicting the appearance of a coating from its microstructure and its constituents.

To identify the key parameters in microstructure characterization and to predict the scattering properties using microstructure data, the research team of the Measurement Science for Optical Reflectance and Scattering project at NIST has been conducting a series of studies on coated materials having various controlled microstructural properties such as surface roughness, pigment size, dispersion, and spatial distribution. Two model systems – a clear coating on a black substrate and a metallic pigmented coating – were selected. Recently, the study of a clear coating model system has been completed and results have demonstrated good agreement between measured and modeled reflectance.[1]

Using the same methodology, we are studying a more complex system – metallic-pigmented coatings. The most commonly used metallic pigments are aluminum (Al) flake. They have a wide variety of flake size and the orientation of the flakes in a coating depends on the processing conditions.[2] Some of the parameters that determine the appearance of Al-flake coated materials are the size and the spatial orientation of the pigments in the coating. In this paper, we will present preliminary results on the effect of pigment size on optical properties using laser scanning confocal microscopy (LSCM) and optical scattering, and discuss the general approach for developing and integrating measurement and modeling efforts to advance the science of appearance characterization.

Experimental

(1) Samples

Three gray, Al-flake pigmented samples provided by E.I. DuPont chemical company[3] were chosen for the study described here. These samples have the same pigment volume concentration (PVC) but different pigment sizes. The sample nominated "C" contains relatively coarse Al flakes (20-30 μm). Figure 1 illustrates a cross-sectional view of sample C. For sample "F" the Al flakes were relatively fine and sample "C-F" contain an equal mixture of coarse and fine Al flakes.

(2) Instrumentation

A Zeiss model LSM510[3] laser scanning confocal microscope was employed to characterize the microstructure of the coatings. LSCM utilizes coherent light and collects light exclusively from a single plane (a pinhole sits conjugated to the focal plane) and rejects light out of the focal plane. The wavelength, numerical aperture of the objective, and the size of the pinhole determine the resolution in the thickness or axial direction (≈ 450 nm in this case).[4] By moving the focal plane, single images (optical slices) can be put together to build up a three-dimensional stack of images that can be digitally processed. The LSCM technique provides a non-destructive, powerful visual aid for charactering the Al flakes in the coatings due to a large contrast in scattering cross section between the Al flake and the polymer binder. In this paper, LSCM images in depth profile or intensity profile presentations are images of overlapping optical slices (a stack of z-scan images) with each z-step from 0.5 μm to 1.5 μm depending on the pigment size and distribution. Acquisition of a typical optical slice (180 μm x 180 μm) takes less than 8 seconds. Various locations of the sample can be scanned in minutes and with two interchangeable incident wavelengths (543 nm and 488 nm).

In-plane bidirectional reflectance distribution function (BRDF) measurements of the coatings were performed using the NIST spectral tri-function automated reflectance reflectometer (STARR).[5] The three functional operations of the STARR facility are absolute measurements of bidirectional, specular, and directional – hemispherical reflectance over the wavelength range of 200 nm to 2500 nm. The incident radiant flux is a monochromatic, collimated, polarized beam with a diameter of 14 mm and bandwidth of 10 nm. For bi-directional measurements, a lens focuses either the collimated incident beam or the image of the front of the sample onto a silicon photodiode. The sample is positioned either in or out of the incident beam using two orthogonal translation stages that determine the incident angle of the beam on the sample and the viewing angle of the photodiode.

Results and Discussion

Image and data analysis from LSCM measurements for the three Al-flake pigmented coatings are presented and compared to the corresponding BRDF measurements.

Figure 2 shows the LSCM images of the three gray Al-flake samples. Note that the first layer of the Al flakes appeared around 60 μm under the air-coating interface and most of the Al flakes are oriented parallel to the surface of the coating. The upper graphs represent the depth profile of the pigment distribution in a series of z-scan optical slices. The dark region indicates the absence of Al flakes in the probed region. The size of Al flakes ranges from 1 μm to 30 μm, and the shape of the flake varies from platelet-like to particle-like. Variations in size and shape of flakes result in different spatial distributions, which cause differences in appearance. For example, Sample C (Figure 2a) having coarse flakes exhibits a grainy finished texture (or "glitter") and Sample F having fine flakes (Figure 1c) exhibits a smoother finish with no noticeable glitter.

From the depth-profile information, we can deduce the near-surface pigment density in the film. For example, the measured maximum depth of sample C is about 27 μm, while the maximum depth of sample C-F and F is only 19 μm in the same illumination condition. This implies sample C has a lower near–surface density, which may introduce higher off-specular scattering. To verify this assumption, we have performed the two-dimensional Fast Fourier transformation (FFT) of the LSCM images to analyze the image in the frequency domain, which will provides the orientation and spacing of the structure in the original spatial (real space) domain.[6] Figure 3b shows the FFT power spectrum of a 2-D LSCM image (Figure 3a) in the frequency domain. Here q is the frequency, which is inversely proportion to the spatial domain size. Figure 3c shows the circularly-averaged magnitude of the FFT power spectrum as a function of q for the three studied samples in Figure 2. In general, the FFT power spectrum is proportional to the scattered intensity and q is related to the scattering angle ($q \sim \sin\theta$), as indicated in the drawing in Figure 3d. To investigate the interaction between light and Al flakes, the circularly averaged scattered intensity from the FFT data of the LSCM images (Figure 3c) were analyzed and compared with BRDF data for these three samples as discussed later in the text. In addition, real-space topography data, as shown in the bottom graphs of Figure 2, can also be used for theoretical scattering modeling using a ray method to calculate the reflectance properties of a coating and compared to the measured BRDF data.

Figure 4 shows BRDF data for these three samples at an incident angle of 45^o and viewing angle of $\pm$ 10 ^{o} with respect to the specular angle. The BRDF data are indistinguishable in the specular (near $\theta = 45^o$) region since all of the samples contained the same polymer binder. However, sample C has a relative higher off-specular (diffusive) intensity than samples C-F and F. The difference in BRDF data is a result of different subsurface microstructure. The FFT data (Figure 3c) exhibits a similar trend as

BRDF data in the off-specular region. Direct comparisons of the BRDF and FFT data are difficult because the viewing area is different in these two measurements. Thus, factors such as the non-uniformity of the Al-flake distribution as well as the artifact induced through image processing could contribute to difference in the BRDF and FFT Data. Nevertheless, this study implies that LSCM images can be used to predict scattering properties. This application will be useful for examining and characterizing the determinant microstructure of products under different processing conditions and for predicting the appearance of the final product using reflectance models developed from microstructure data.

Summary

We have demonstrated that laser scanning confocal microscopy is a powerful technique for characterizing coating microstructure. The microstructure of Al-flake pigmented coatings was easily obtained using LSCM. Using FFT image analysis, the microscopic data can be related to the optical scattering measurements. In addition, real-space topography data or 3D reconstruction images can be used for scattering modeling to calculate the optical properties, and be compared to the measured BRDF data.

Acknowledgement

This research is part of the Measurement Science for Optical Reflectance and Scattering project at NIST. For more information, please visit the Appearance Project web site, http://www.ciks.nist.gov/appmain.htm.

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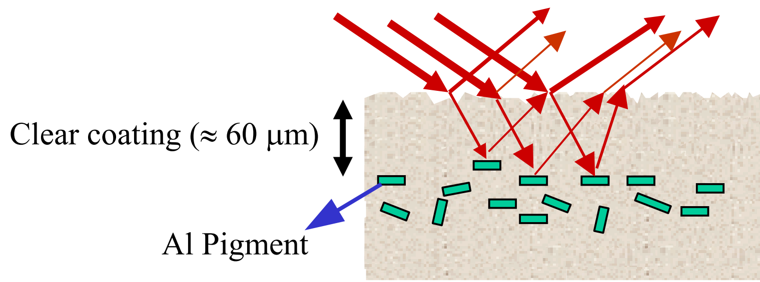

Figure 1. A cross-sectional view of an Al-flake pigmented coating. The primary (first-order) interaction between light and the pigment is the specular reflection as illustrate in the graph.

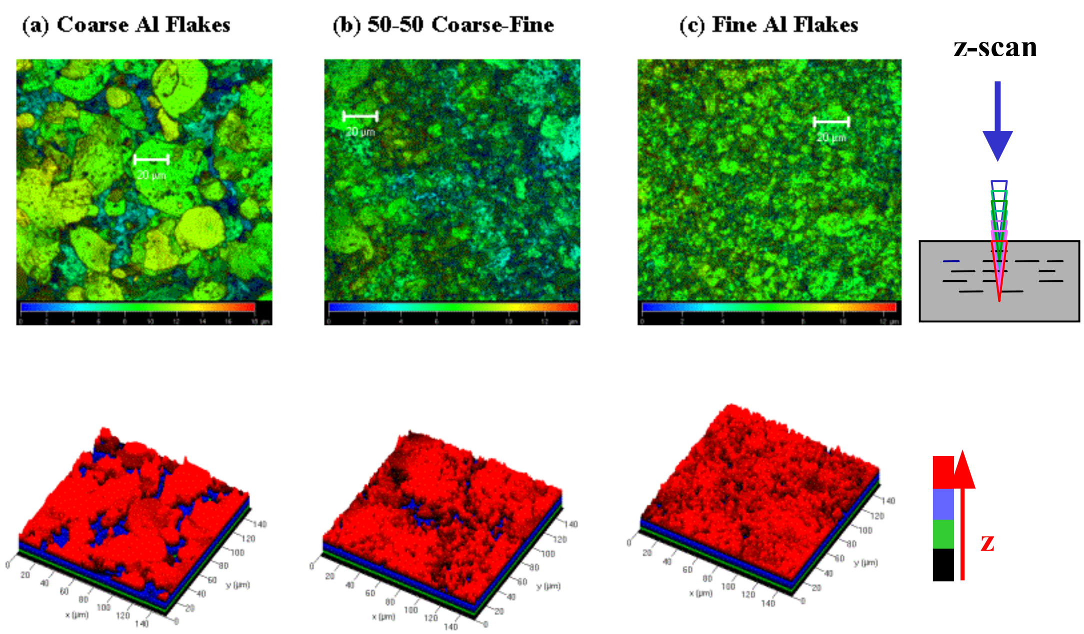

Figure 2. LSCM images of three gray, Al-flake pigmented coatings having same PVC but containing different pigment sizes, (a) coarse, (b) 50 % coarse with 50% fine, and (c) fine Al flakes. Upper graphs show the LSCM in the depth presentation in a typical z-scan; the color blue represents the pigment is far away from the top surface and color red is near the top surface. Bottom graphs show the topographic presentation of the same images for theses three samples.

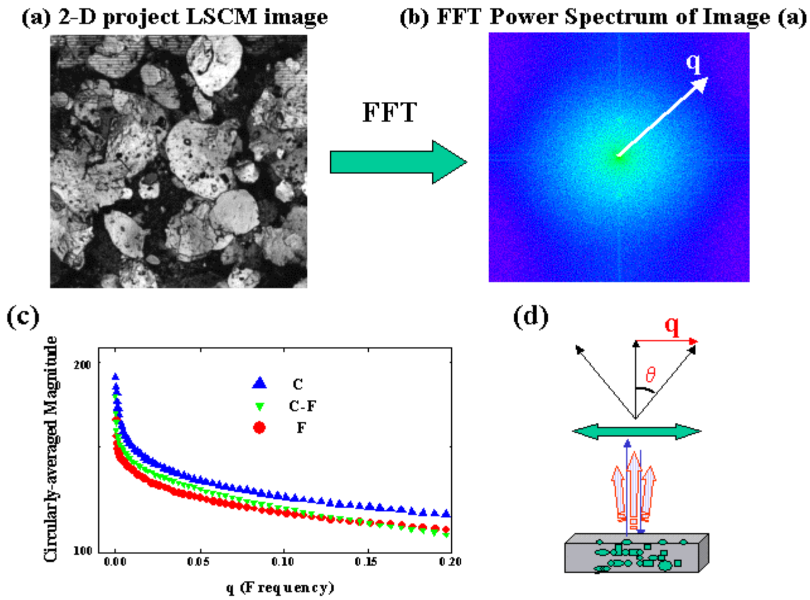

Figure 3. (a) 2-D projection of the LSCM image. (b) FFT power spectrum of image (a) (c) Circularly-averaged magnitude of FFT power spectrum as a function of frequency q. (d) A drawing illustrates the relationship between q and scattering angle.

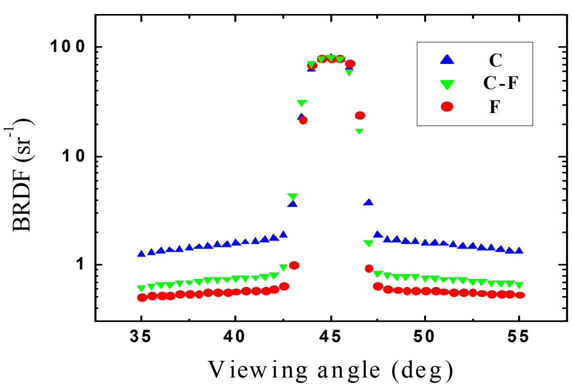

Figure 4. BRDF data of coarse(C), mixture of coarse and fine (C-F), and fine (F) Al-flake pigmented coatings at an incident angle of 45° and viewing angles of ± 10° with respect to the specular geometry. The expanded uncertainties are less than ± 0.5 % (k=2) and the size of the error bar is smaller than the size of the symbols used in the graphs.