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Relation between optical non-contact profilometry and AFM roughness parameters on coated papers with oil-filled nanoparticles

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ABSTRACT

In parallel with the development of nanoparticle coatings for protection of paper substrates, detailed descriptions of the surface topography with micro- to nanoscale roughness features are needed. In this work, papers have been coated with poly(styrene-co-maleic anhydride) nanoparticles including different types of vegetable oils and the surface roughness was evaluated at 2000 × 2000 μm^2 to 0.2 × 0.2 μm^2 length scales by combining non-contact optical profilometry (NCP) and atomic force microscopy (AFM). The relationships between roughness data were studied for statistical roughness parameters, spatial roughness parameters and in the frequency domain. In order to compare AFM roughness more accurately, the original images were flattened to remove effects of the underlaying fibrous substrate and highlight features of the nanoparticle coating. More detailed information on the coating topography could be obtained by considering bearing ratio curves and histograms, where it was concluded that the oil-filled coatings form a rather thin and continuous coating that closely follows the shape of the cellulose fibers. The relation between statistical roughness parameters from NCP and AFM follows an exponential trend with relatively low coefficient of determination. The increase in surface roughness with length scale showed a transition point attributed to short- and long-range surface features. Therefore, the correlation length was used as a spatial roughness parameter that provides a successful extrapolation of the average roughness over different length scales in a double logarithmic diagram with very high coefficient of determination. Based on the power spectral density, it was difficult to exactly distinguish between the different types of SMI/oil coatings, as they include similar nanoscale features. The frequency roughness parameters were better suited for extrapolation than statistical roughness parameters but little less efficient than the spatial roughness parameters.

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1. Introduction

The surface topography of paper determines its environmental interaction and fundamental properties of tribology, absorption, optics, adhesion, wettability or biocompatibility. The latter are directly related to technological applications and processing of paper sheets with specific requirements of gloss [1], printability [2,3], water repellency [4], or runability [5]. The paper surface quality is generally described by two phenomena: (i) the waviness, referring to large-scale deviations from a flat sheet such as so-called piping streaks due to different fiber alignment across the paper machine, (ii) the roughness, defined on a

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smaller length scale with high-frequency surface irregularities. Approaches for monitoring paper surfaces that distinguish between both length-scales are used for statistical process control [6]. Otherwise, the macro-/microscale roughness features correspond to those with dimensions larger/smaller than the wavelength of optical light. The modeling of these multi-scale roughness features might give a good description of reflectance properties [7].

The complex surface topography of paper substrates depends in first instance on the ordering of the cellulose fibers causing a porous paper structure with intrinsic roughness levels [8,9], attributed to (i) the basic fiber dimensions with internal microscale fibrils, (ii) the mesh of the forming wire with dimensions of about one millimeter dimensions, and (iii) the macroscopic mass distribution of the fibers. As a result, the macroroughness of a paper sheet results from fiber interactions, poor dispersion, ionic destabilization or flocculation. Depending on its application, the topographical properties of paper surfaces are optimized through a water-based pigmented coating. As such, the microroughness of paper surfaces depends on pigment particle size distribution, particle shape, shrinkage of the coating during drying, coating hold-out, coating weight and binder. The further processing of sheets may induce fiber rising and consequent surface roughening [10]. It is evident that the base paper characteristics such as roughness, absorbency and sheet matrix formation will influence the final properties of the coated sheets: an increase in base paper roughness gives lower smoothness and gloss to the coated paper and often, the structural deficiencies of the base paper are enhanced after coating [11]. Depending on the coating procedure, curtain and blade coated papers induce different roughness profiles [12,13]. In recent years, nanoparticle coatings have been developed in order to specifically increase the surface hydrophobicity [14,15]. The nanoparticle deposits cause an additional roughness at the nanometer scale, which further broadens the need for characterization of paper coatings at different scale lengths.

In order to provide a good description of the surface topography, papers have been characterized by different techniques such as profilometry, interferometry, chromatic aberration, microscopy, air-leak tests, speckle patterning [16,17]. In parallel, on-line surface roughness techniques are based on light triangulation [18]. Topographical surface maps can be obtained with optical interference based on the variation of focus over the surface [19]. The white-light interferometry has been implemented for rough surfaces [20]. However, optical measurements of microroughness can only be compared for substrates with similar macroroughness: the microroughness may be underestimated for substrates with high macroroughness, due to directional-diffuse scatter of the light intensity; typically, a limiting root-mean-square microroughness of 50 nm is reported [21]. The optical reflectometers also simultaneously measure macro- and microscale roughness in combination with gloss [22]. Atomic force microscopy (AFM) has become a standard technique for assessing sub-micron structures on paper coatings and sizing [23,24]. By comparing roughness from AFM and non-contact profilometry on glass–ceramic substrates, a methodology for selecting scan sizes and sampling intervals was suggested [25]. A comparison of AFM and optical roughness for calcium carbonate coatings shows that differences were mainly related to the effective "probe" diameter of both techniques: the optical roughness measurements are biased to roughness features somewhat larger than the AFM tip [26]. The effects of long-range and short-range roughness from AFM and optical profilometry could be distinguished for kaolin coatings [27]. A low-coherence interferometer has been used with spatial resolution of about 15 μm, but much higher vertical resolution [28]. Measurements by confocal laser scanning microscopy (CLSM) could be done by fluorescent staining, but reacted selectively with some components of the coating [29]. By comparing AFM and CLSM [30], it has been concluded that local tilt and flattening procedures in AFM images highly influence the local roughness. New advances were made in the 3D characterization of coated papers by introducing laser profilometry and X-ray microtomography [31].

As paper profiles are stochastic in nature, a set of statistical roughness parameters can be calculated as different moments of the height distribution (mean height, standard deviation of surface heights, skewness, kurtosis). However, the single numbers cannot fully describe a surface topography and an additional set of spatial roughness parameters has been introduced based on the lateral distribution of surface heights. The roughness parameters of a platy kaoline clay coating (600 nm particle sizes) have been studied by a two-point correlation function where both amplitude and lateral spacing of the surface heights were considered [32]. In the frequency domain, differences in coating structure or fiber composition were visible in the roughness power spectra [33]. On the other hand, the roughness values depend on the length scale related to the bandwidth of the studied physical features. Therefore, the roughness values strongly depend on the evaluation length [34] and discretization step [35]. Finally, 11 roughness parameters have been systematically studied at different magnifications [36]: based on a multi-scale analysis, a method of fractal analysis has been proposed [37].

In view of depositing organic nanoparticles with encapsulated vegetable oils as hydrophobic paper coating, a good understanding of the roughness at different length scales is needed. From the different basic principles of optical roughness profilometry and atomic force microscopy, agreement between both cannot readily be expected and a detailed surface evaluation should be based on a description of statistical and spatial roughness parameters that allow extrapolation between different scale lengths.

2. Experimental details

2.1. Paper coating formulations

Organic nanoparticles were formed by imidization of poly(styrene-*co*-maleic-anhydride) in presence of ammonium hydroxide, resulting in an aqueous suspension of poly(styrene-*co*-maleimide) or SMI nanoparticles. During the imidization reaction, different vegetable oils were added resulting in hybrid organic nanoparticles with soy

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