Profilometry of PE-coated paperboard using chromatic confocal microscopy and 3D SEM stereo-photogrammetry

J Brugés Martelo[1], C Liguori[2], M Andersson[1], H Andersson[1] and J Thim[1]

[1] Electronic Design Department, Mid Sweden University, Holmgatan 10, 851 70 Sundsvall, Sweden

[2] University of Salerno, via Ponte Don Melillo, 84084 Fisciano (SA), Italy

E-mail: `javier.bruges@miun.se`

Abstract.

In food packaging, polyethylene(PE) coating is applied to paperboards to act as a functional barrier and to provide smoothness to enhance printability. These characteristics are related to the surface and optical parameters that are monitored during the manufacturing process. Surface roughness belongs to these sets of the monitored parameters and it is of most importance for the functional and optical quality of the product. Measurement of surface roughness using optical profilometry has gained importance in paper industry. However, visible light-material interaction is complex on paper and paperboard and the spatial resolution of the instruments is limited by the wavelength of ligth. A Scanning Electron Microscope (SEM) is an alternative to overcome the limitation on spatial resolution and the use of stereo-photogrammetry on the SEM images can provide the topographic datasets. We measure the surface topography of high-quality PE coated paperboard using an SEM stereo-photogrammetry and a Chromatic Confocal Microscope (CCM) and we compare the surface roughness statistical parameter Sq and calculate the 1D Power Spectral Density ($1D$-PSD) function from both measurements. To present in this article an accurate correlation between the measurements, we exploited the spatial bandwidth limitations of the instruments and used the new ISO 25178 as a guideline for the measurements. We found close agreement between calculation of statistical parameters and we could identify the effects of applying a non-standard topographic measurement like the SEM stereo-photogrammetry. In conclusion, we can use in the future SEM stereo-photogrammetry as a method to characterize surface roughness of coated paperboard and compare with other optical techniques.

1. Introduction

In the paper industry, paperboard grades are differentiated depending on its functionality, being packaging boards within the higher in optical and surface quality. Quantifying the effect that surface roughness has on paper and paperboard, e.g. gloss uniformity and optical quality, it's of most importance to assess the difference between paperboard grades and can be measure along the manufacturing process [1]. Polyethylene(PE) coating is applied on paperboards for its use in food packaging products due to its excellent barrier functionality against moisture. For PE coating, the surface roughness is used to control smoothness affecting printability and it is measured by the industrial standard air leak methods, i.e. Bendtsen, Bekk and Parker

Print Surf (PPS) [2]. These instruments estimate surface roughness by the correlation between the rate at which air escapes from a system formed by metal plates sandwiched between the paperboard. These systems are calibrated to provide roughness values for a limited range of surface spatial distribution. Although a good agreement between paper and paperboard grades and the surface parameters is well defined, these methods are not capable to provide detailed information of surface texture or spatial information, e.g. roughness variation, associated with the quality of the product.

In a recent study, a set of optical profilometers have been compared after measuring roughness parameters on various paper grades[3]. In the visible spectrum, light-material surface interaction is complex, depending on both material composition and surface topography, making it difficult to establish a common technique to characterize all grades. Optical devices such as confocal scanning microscopy [4] and scanning electron microscope (SEM) [5] are valuable tools for surface characterization. A chromatic confocal microscope (CCM) is a variation of the former instrument and it has been included in the new ISO 25178 [6] for areal surface characterization. Large areas of paperboard can be analyzed by the XY coordinate measuring scanning set-up with a lateral accuracy of one micron and depth resolution of tens of nanometers [3]. On the other hand, SEM is commonly used for paper characterization, where imaging the sample cross-section can reveal the effects that local structures in the material composition have on different surface parameters. Cross-section micrographs of paper and digital image analysis has been applied to obtain relations between the base sheet distribution and the coating thickness uniformity [?] and has been used to quantify different surface descriptors, including surface roughness, of commercial super-calender paper [7]. Although these methods provide good agreements of the local behavior affecting the paper composition, they are not suitable for mapping large areas within the sample. Besides, preparing a cross-section for these analyses requires extensive sample preparation and expertise. However, the high dynamic range of the SEM is an advantage in comparison to other optical scanning systems and gives the possibility to image specimens using a larger field of view within a higher depth of field and providing surface details up to nanometer resolution. Besides the highly detailed topographic images acquired by the SEM, the images are essentially two-dimensional and the grayscale information does not represent the height distribution of the specimen. Detectors providing high resolution images are available in the newer SEMs and software developers are now integrating stereo-photogrammetry software for metrology applications [8], for instance the MeX software from Alicona that is used in this article. Photogrammetry is a technique used to estimate height information from two-dimensional images using one or several images. To overcome the limitation of the SEM to acquire accurate topographic information, several photogrammetric techniques have been proposed, where the stereo-photogrammetric technique is one of them [9]. Stereo-photogrammetry uses two or more stereo-pair images acquired from different perspectives of the same scene. Most sample chambers found in nowadays SEMs are capable of orienting the sample towards the detector at different tilting angles. Images from an area in the sample can be acquired at different perspectives from the detector. Profilometry using SEM to characterize paper samples has been reported by Enomae [10]. Using two detectors positioned at both sides of the electron beam column can acquire the stereo pair images of the paper samples and the reconstruction of the surface texture.

Recently comparisons on optical coherence scanning interferometer and 3D stereo-photogrammetry from SEM stereo-pair images for dental implants [11] have opened the path to an investigation in other fields (*from now in this article we refer as SEM stereo-photogrammetry*). Statistical parameters for characterizing surface topography are of primary importance for roughness analysis, e.g. RMS roughness and the power spectral density function (PSD). PSD analysis is used for instrument inter-comparison, using the spatial bandwidth limits of the instruments as the integration limits for surface roughness calculations and to highlight the differences in the results. Using two different profilometers with overlapping spatial bandwidth distributions, it was possible to study a larger scale of PSD on the topography of paper samples [12]. Different paper grades and the knowledge of bandwidth limits of the instruments were used for the study. Using a similar approach to obtain scale-limited surfaces within a finite range of spatial distribution, a multi-scale analysis using a focus variation optical profilometer is applied to different paper grades and a set of statistical parameters is analyzed to assess its performance along the different bandwidth limits [13]. For a good metrological practice, a guide for instruments inter-comparison has been published and the technique of bandwidth matching has been formally introduced [14]. The type of scanning systems that projects a beam spot on the sample, the beam size defines the spatial resolution of the instrument and the spatial bandwidth limits of the measurement. In order to carry out comparative instrumental studies, matching the number of pixels on the area of comparison is necessary. The CCM and SEM spatial resolution is limited by this beam spot characteristic.

This article compares the surface topography of a commercial high-quality PE coated paperboard sample from the profilometry measurements acquired by using a chromatic confocal microscope and a SEM stereo-photogrammetry technique. We compared statistical roughness parameters, i.e. Sq, and we used the 1D PSD from machine direction (MD) and cross-machine (CD) direction [15], to calculate the roughness parameter associated with its curve in both datasets. The following section will describe the sample preparation, the instruments involved in the measurements and the methods for the surface parameter extraction. Following is the introduction of the topography maps and the result from the metrology analysis. Finally, conclusions and future work are presented.

2. Experimental

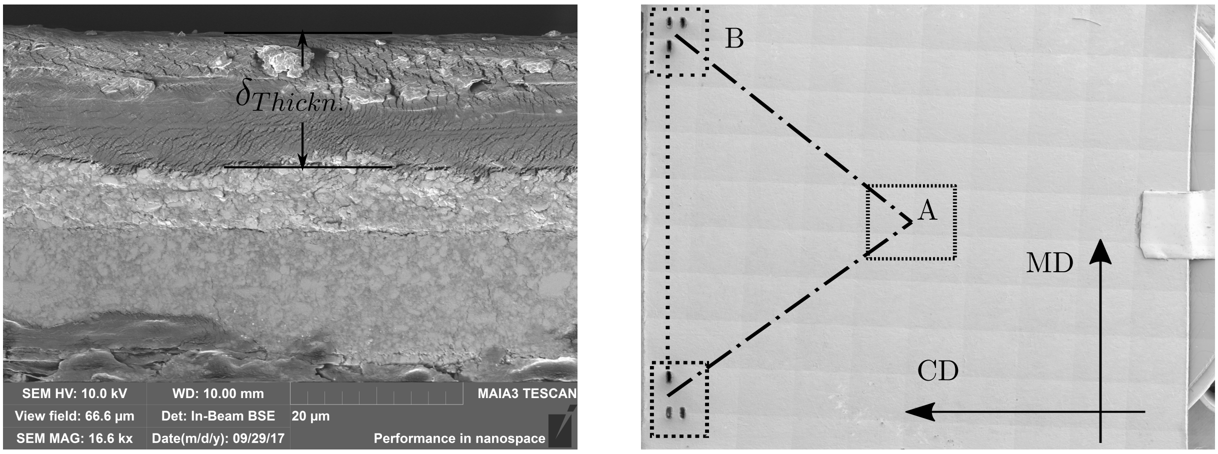
In this section, the sample preparation and instruments selection are presented. Further, we described the choice on the statistical parameters used for the analysis and comparison of the topographic datasets.

2.1. Material

The sample selected for this study is a 20 mm by 20 mm piece of low-density polyethylene (PE) coated paperboard. The thickness range, δ_{Thickn}, of the PE on the paperboard (figure 1(a)) ranged from 13 μm to 15 μm. We studied the topography of the sample from the top (figure 1(b)), where area (A) was selected as the region of interest (ROI) in both CCM topography measurement and SEM stereo-pair images. A pattern (B), engraved using a CNC laser machine, aided to identify faster the ROI of the scanned surface area. The pattern also provides information regarding the orientation of the sample during the manufacturing process, i.e. machine direction(MD) or cross-machine direction (CD) that are important when measuring statistical parameters on anisotropic surfaces like those found in paper and paperboard. The orientation of the fibers is found to be related to the gloss uniformity and the surface macro-roughness. It was necessary to apply a metal coating on top of the sample in order to get high contrast images from the SEM. A 3 nm layer of Iridium coating was selected due to the smaller grain size and even coating distribution that this metal provides in contrast to more commonly used materials, e.g. gold. All measurements presented in this article use the same PE paperboard coated sample with a layer of Iridium.

2.2. Instruments

Two topography datasets from the same sample were obtained from two different optical instruments, i.e. a Chromatic Confocal Microscope (CCM) and a Scanning Electron Microscope (SEM). The two mentioned instruments focused a beam at the surface of the sample and it is scanned in a raster strategy along the sample, while in the CCM the sample is positioned on a



(a) (b)

Figure 1. (a) The SEM cross-section image showing the material distribution paperboard sample. δ_{Thickn} shows the PE coating with a thickness range of 13 μm to 15 μm. (b) The top view from the $20x20mm$ paperboard sample. A marking system helps to identify the center of the sample and its orientation according to the manufacturing process, i.e. MD and CD.

stage that is moved transversally from the optical axis and on the SEM the sample is stationary during scanning process while the electron beam is steered along the sample by deflecting the beam with a set of magnetic lenses. Due to the high depth of focus under a large field of view, SEM images can be used as an analytical topographical tool for surface analysis, but its grey scale images cannot be directly converted to a height map dataset. In contrast, CCM is a true areal measurement system that provides direct topographic data on the flight and this method is included in the new ISO 25178-602 [16].

Finally, the stereo-pair images are combined by means of a stereo-photogrammetry in order to obtain the three-dimensional dataset.

2.2.1. Chromatic Confocal microscope During this experiment, we used an FRT MicroProf (Fries Research & Technology GmbH; Bergisch Gladbach, Germany) placed in a temperature controlled room. Measurements were taken from one of the sensors on the instruments, i.e. H0 chromatic probe, with a calibrated depth range of 300 μm and lateral resolution (beam spot size) of 1 μm. Being a confocal microscope type, the CCM (figure 2) uses a broadband light source and a spectral detector arranged in a geometrical matching of its focal point conjugates at the image space. The difference of this instrument from all other types of confocal microscopes is that it employs an objective with a high axial chromatic aberration designed specifically to be long and known. A color-coded depth information can be estimated from the light reflected at the object's surface with nanometer accuracy without the need of a vertical moving stage on the confocal system. Light scattered or reflected back to the pinhole-detector is calibrated against the height and the intensity values from the light reflected back into the system that is analyzed by a spectrometer. The grating spectrometer provides instantaneous height information making these types of devices much faster than traditional confocal microscopes. The range of heights that this instrument can measure is limited by the ability of the dispersive objective to decompose the light and the light source spectrum. Another drawback is the limitation to measure objects with steep local variations.

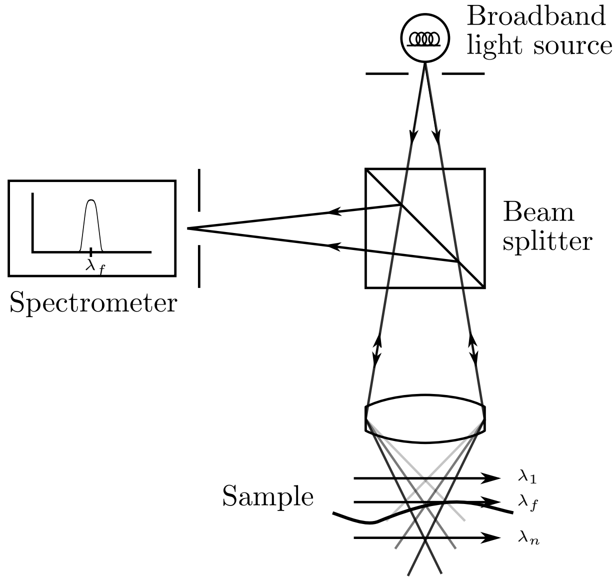


Figure 2. Chromatic confocal microscope.

2.2.2. Scanning Electron Microscope SEM images were taken from a TESCAN MAIA3 GMU (TESCAN Brno, s.r.o.; Brno, Check Republic). Multiple detectors are available on an SEM and for pure topographic imaging, a secondary electron detector was chosen. Electron beam voltage of 2kV was selected which correspond to a beam spot size of 25 nm. In order to create stereo-pair images, eucentric tilting of the sample's stage was used with a tilting of ± 5 deg. We chose an initial tilted position of 20 deg of the sample stage towards the detector to increase electron yield sample-detector interaction and to obtain higher topographic contrast on the images. Before scanning the surface, it is necessary the co-localization of the region of interest. Using the CCM 2D image to aid the co-localization and MeX software co-localization tool assured the correct positioning of the sample. For all images a magnification of 554x was selected, resulting in a field of view of 2 mm, where later a cropping function selecting the region of interest for instrumental comparison was applied. Images of 4096x4096 pixels with 488.28 nm lateral resolution were necessary to obtain in order to increase the topographic information that the photogrammetric step requires.

2.3. SEM Stereo-photogrammetry

Stereo-pair images from two perspectives are used to generate a disparity map (figure 3), i.e. features recognizable in both images are used to measure axial distance, and by means of triangulation, the height information can be calculated. We used the software MeX 6.0 (Alicona Imaging GmbH, Raaba/Graz, Austria) to create the topographic dataset from the selected sample. A downscaling of the generated topographic dataset using the bilinear interpolation offered by the software was necessary to apply in order to match the lateral resolution of the CCM topographic dataset.

2.4. Statistical parameters

To obtain scale-limited surfaces of both topographic datasets, the general procedure presented in the ISO 25178-3 [17] was followed. A second order polynomial regression was applied in order

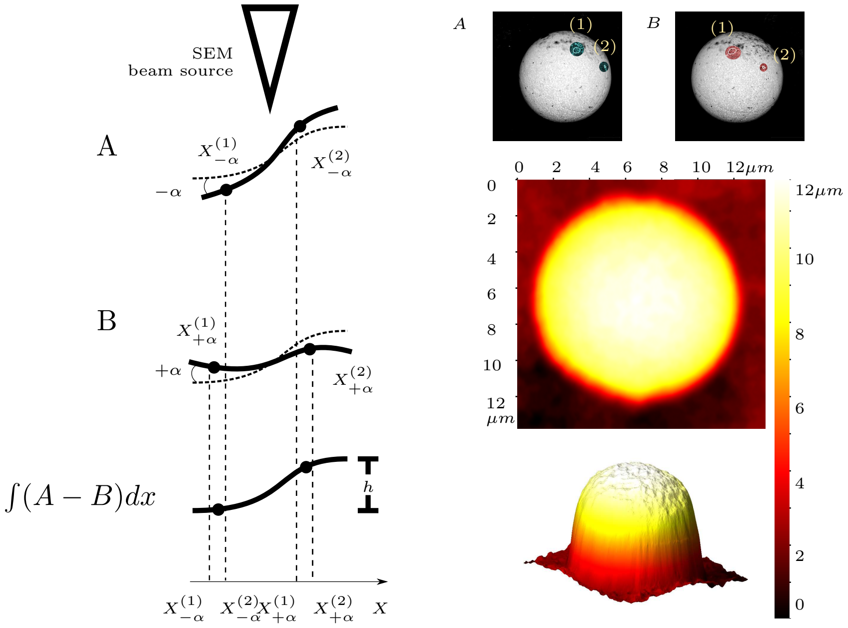


Figure 3. A stereo-pair photogrammetry technique requires at least two images from different perspectives, i.e. A and B. Image pixels in both images undergo a lateral displacement, e.g. (1) and (2). By knowing the distance that identified features are displaced and the angle of perspective $\pm\alpha$, triangulation is used to calculate the height in every pixel.

to remove the form from the extracted surfaces. S-filter and L-filter with nesting indexes $2,5\mu m$ and $250\mu m$, respectively, and Gaussian characteristics were selected to generate the scale-limited surface. The lateral resolution of the CCM dataset, i.e. $1,36\mu m$, was used as the minimum possible value for the S-filter nesting index. Each extracted dataset used for this comparison had the same number of pixels and lateral resolution, important for the bandwidth matching condition. The SL-surface generated was conformable with the bandwidth characteristics of the topographic techniques.

Areal parameters introduced in the ISO 25178-2 are used as a tool to analyze surface texture and its functionality. The root mean square surface heights (Sq) and the 1D power spectral density (1D-PSD) function in the axial directions, i.e. CD and MD direction, were calculated from the obtained SL-surface. Sq served as a comparison tool for the analysis of the overall areal roughness. However, it does not take into consideration directional features like those encountered on anisotropic surfaces. The 1D-PSD is used within the spatial bandwidth limits and the integration of the curves provides a direct relation of the roughness:

$$PSD(f_x) = \frac{\delta_x}{N}|FFT|^2$$

,

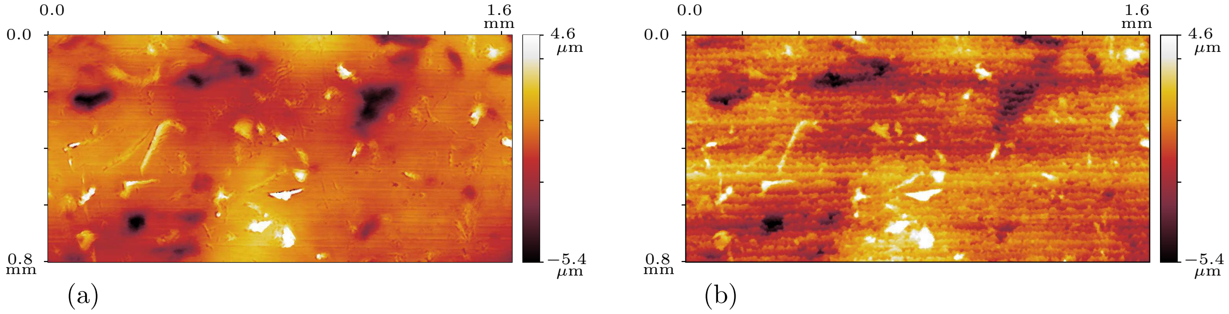


Figure 4. Primary surface of (a) CCM and (b) SEM. S-filter with $2,5\mu m$ nesting index was applied and second-order polynomial regression removed the form.

$$\sigma_{rms}^2 = \int_{f_{min}}^{f_{max}} PSD(f)df \tag{1}$$

where δ_x is the lateral resolution of the system, N the number of pixels in the direction of measurement and $|FFT|^2$ is the fast Fourier transform of the SL-surface. By integrating the PSD we can obtain the profile parameter σ_{rms} which can be compared for CD and MD direction.

3. Results and discussion

A close agreement was found between the two instrument measurements where profile features and overall height distributions are recognizable in both dataset representations (figure 4). Nevertheless, it is clear that the topography obtained with the SEM stereo-photogrammetry technique suffers from artifacts that are formed along the axis of tilting that coincide with the CD direction of the paperboard manufacturing process. Many flat areas encountered on the surface and the low magnification of the SEM images, which affects the parallax photogrammetry condition to estimate the disparity map, amplified these errors. This is a known drawback of this photogrammetric technique [18].

A set of 100 height profiles along the CD in both profiles were extracted and averaged (figure 5). Normalized root mean square deviation from the curve profiles deviate from each other about 6%. This result suggest that the level of error induced by the artifact can be minimized by improving the SEM stereo-photogrammetric technique or applying filters to minimize the effect of the deviation.

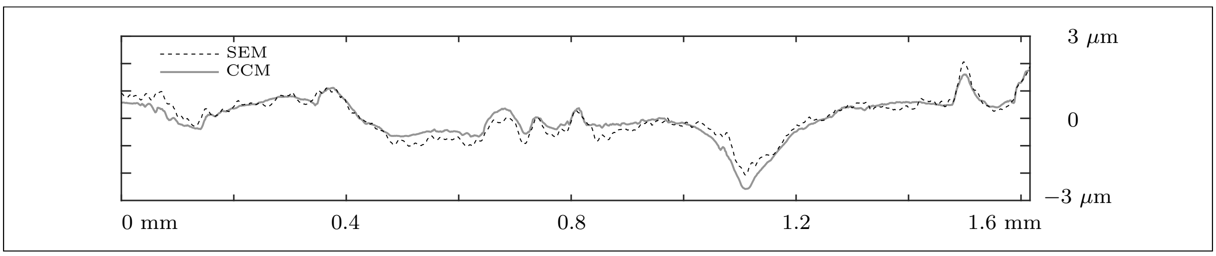


Figure 5. Recorded profiles from both topographic datasets obtained after the F-operator is applied.

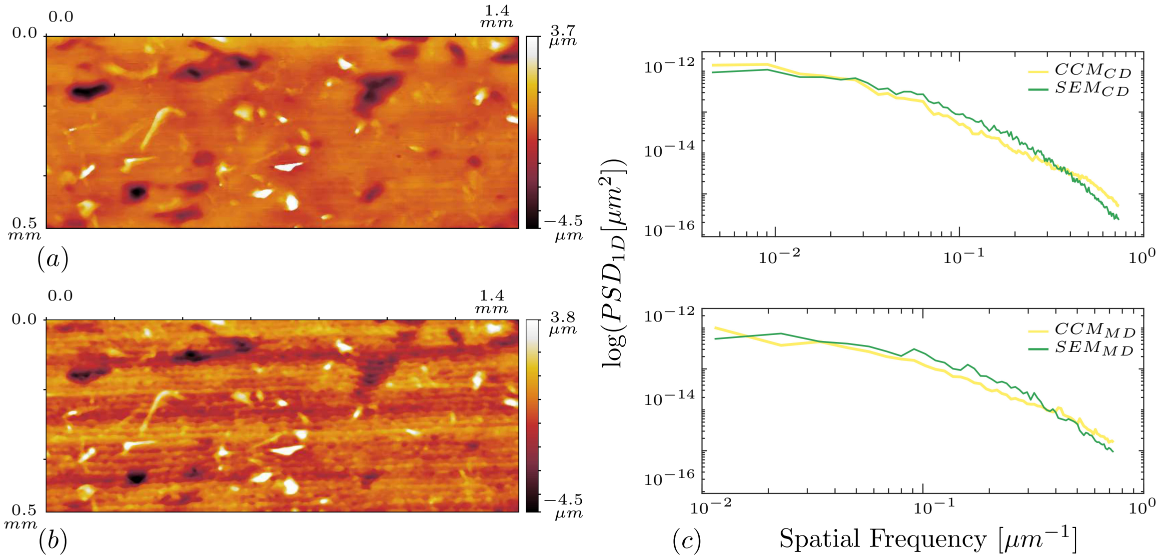


Figure 6. Roughness component after SL surface obtained from (a) CCM and (b) SEM stereo-photogrammetry topography datasets. We calculated the PSD from both topographies (c) in the CD and MD direction to obtain a direct comparison of the orthogonal texture behavior

From the SL surface, we extracted the roughness component in both datasets and the 1D PSD in both directions can be calculated (figure 6). Table 1 contains the resulting areal parameter Sq and the roughness value obtained after integration of 1D PSD curves from equation 1, both in MD and CD. The artifacts present in the SEM stereo-photogrammetry topographic dataset increases the roughness values. However, close similarity is found between the statistical parameters from both instrument results and the significant difference associated to the areal parameter Sq is close related to the increase value of $\sigma_{MD}[nm]$ that correspond to the direction orthogonal to the perspective projection of the SEM stereo-photogrammetry technique.

Table 1. Calculated statistical parameters: Sq from areal topography and σ_{rms} from the 1D PSD in MD and CD.

Instrument	Sq $[\mu m]$	$\sigma_{CD}[\mu m]$	$\sigma_{MD}[\mu m]$
SEM	$1,07$	$1,05$	$1,14$
CCM	$0,97$	$1,11$	$0,94$

4. Conclusion

Statistical parameters, like the surface roughness of PE-coated paperboard, can be obtained from direct and indirect areal measurement techniques, e.g. chromatic confocal microscope and stereo-photogrammetry based on SEM stereopair images respectively. We showed that measurement comparison between the two different techniques is possible when bandwidth matching condition is ensured within the spatial limitation of both techniques. Artifacts present in the SEM stereo-photogrammetry topographic dataset where exposed in the image topography but the calculation of the statistical parameters allows us to identify the relations between the instruments. We believe that the photogrammetry technique can be improved if further constraints in the parametric calculation, e.g. parallax condition and surface features height estimation, are included during the stereopair image acquisition.

Since the SEM stereo-photogrammetry requires much effort from the operator and the quality of the results depends directly on his/her level of expertise, this technique will still remain as an alternative for paperboard surface characterization. In the future, we will use these techniques and work at the application level, where functional surface parameters from paper and paperboard are going to be investigated and compared to other paper characterization reference methods.

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