

New concepts for 100% inspection of coated and surface treated webs

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Hyperspectral imaging (HSI) is an ascending and promising technology for challenging inspection tasks. Initially developed for remote sensing applications, the HSI technology has spread to different areas of applications over the last years. One of them is the 100% inspection of coated or surface treated webs.

HSI combines spectroscopy and imaging and provides high-resolution data in the spatial and the spectral dimension simultaneously, containing much more information than classic line-scanning cameras. Therefore it allows the analysis and evaluation of spatial differences of chemistry, morphology and topology. The information content of the hyperspectral measurements is outstanding in contrast to monochrome and RGB-imaging (like in standard machine vision inspection) on the one hand, and to normal single-point spectroscopy on the other hand. The high dimensional ‘image stack’, acquired by HSI reveals completely new possibilities for data evaluation and consequently for process monitoring tasks. The spectral information is combined with the information on texture, topology, shape and structure of up to 1000 images. HSI is commonly suitable for UV, VIS and NIR spectral range (250-2500 nm).

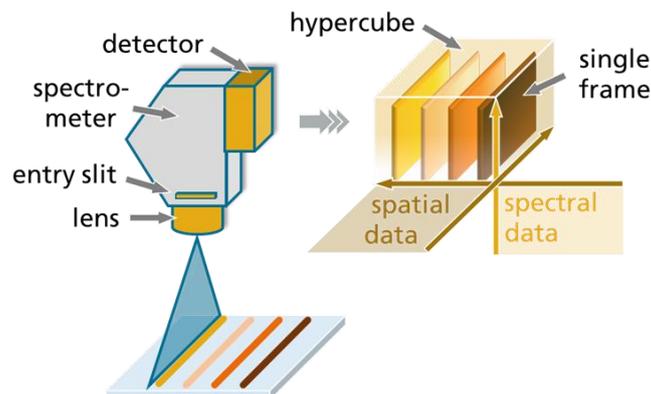


Figure 1 Scheme of the „pushbroom“ HSI technology

One of the important details in hyperspectral imaging is an appropriate lighting and referencing. Whereas spot lighting can cause shadowing and is in thin film applications fragile due to primary beam reflections, spots and substrate behaviour, a diffuse, homogenous lighting is the right choice for thin film and web monitoring. To bring this requirement into a line (as it is necessary for the so called “pushbroom imaging”, see Figure 1) is challenging. In addition the lighting must be broadband emitting for the spectral data recording.

For this purpose, the Fraunhofer IWS has developed an integration tube, made of an optical PTFE, with a very high reflection of > 98 % for the whole spectral range. In combination with further self-designed power sources a continuously variable power level can be adjusted for intensities up to 29 klux for the covered line. The homogeneity of the diffuse lighting tube was determined at > 95 %.

Hyperspectral thin film imaging can be done on two ways: The hard modelling approach calculates the target parameters of the layer stack (e.g. layer thickness) by using physical models whereas the soft modelling approach corresponds to statistical data treatment and chemometric data evaluation as well as to prediction and regression models.

Using the hard modelling approach, the reflectance (or transmittance) of a coated substrate can be calculated on any sample point based on an optical model including the parameterized optical functions (CAUCHY-model for the refraction index n and the absorbance k)¹ using the FRESNEL equations and transfer matrix formalism.² In result a distribution of the desired parameter can be given (Figure 2).

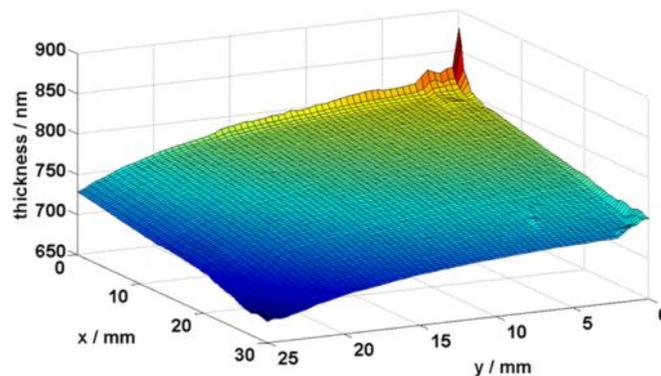


Figure 2 Thickness distribution of a Diamor® (diamond-like-carbon, DLC) thin film

As an example, an Al_2O_3 layer on a stainless steel foil should be monitored regarding the layer thickness and defects. The layer itself was deposited by a continuous slot-die coating process and is a catalyst layer for the growth of single wall carbon nanotubes (SW-CNTs), a homogenous layer is mandatory for the wanted end use in a battery application. While the calculation by hard modelling is very time-consuming, especially for larger sample sets and a possible inline application, a soft modelling approach was used for the inspection of the aluminium oxide layer.³

First, reference samples were measured by ellipsometry and hyperspectral imaging. Then the real layer thickness (outcome of the ellipsometric measurements, Table 1) and the pre-processed and averaged HSI spectra were used to build a PCR (principle component regression) and PLS (partial least square regression) model as well (Table 2). The PLS model shows a slightly higher regression quality (R^2) of 0.996 and was used for the further

data evaluation. This step builds the training data set (according to layer thickness) for the subsequent prediction. The prediction step can be done easily within a process monitoring.⁴

Table 1 Layer thickness and Cauchy-parameters of the Al₂O₃- layer determined by Ellipsometry

<i>parameter</i>	<i>thickness / nm</i>	<i>A_n</i>	<i>B_n</i>	<i>C_n</i>	<i>A_k</i>	<i>B_k</i>	<i>C_k</i>
	52.4 – 66.2	1.5827	0.0191	-0.0013	0.1359	0.3828	400

Table 2 PLS and PCR model for the prediction of Al₂O₃ layer thickness

<i>model</i>	<i>No. of latent variables</i>	<i>R²_C</i>	<i>RMSEC</i>	<i>R²_{CV}</i>	<i>RMSECV</i>
<i>PLS</i>	4	0.996	1.5	0.979	3.6
<i>PCR</i>	4	0.992	2.0	0.978	3.7

Further measured hypercubes undergo the same pre-processing and all spectra were used for prediction of the Al₂O₃ layer thickness. In result a thickness distribution for the full sample area can be given. But to avoid an individually optical inspection of every thickness map, quality control parameters must be introduced. Such well-defined parameters are important for the integration of the HSI as a monitoring tool into industrial processes. According to the requirements in this example, the mean layer thickness and uncoated area are suitable for the evaluation of the samples (Figure 3).

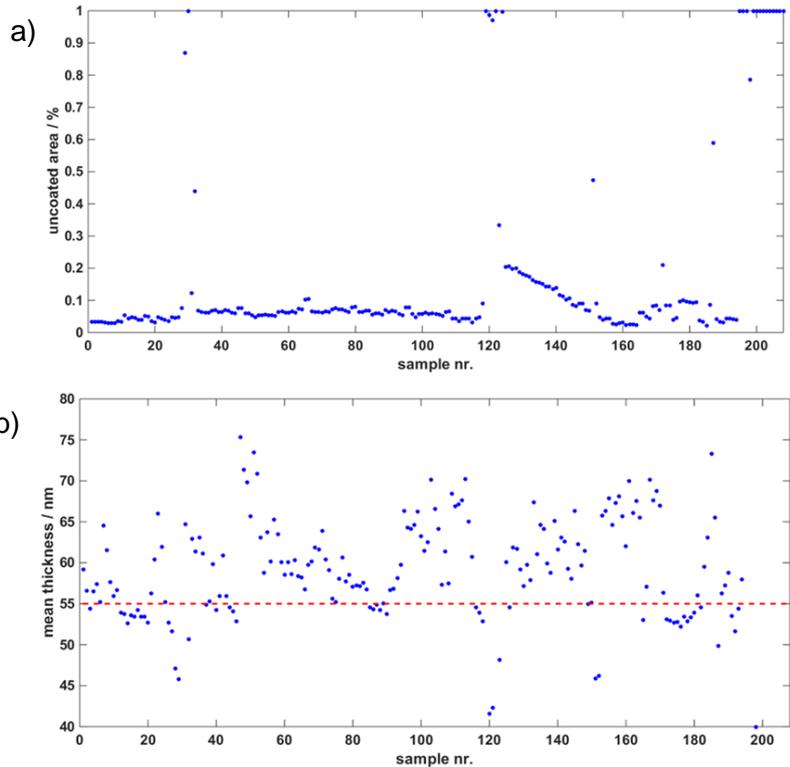


Figure 3 Quality control parameters of the Al₂O₃ layer; a) uncoated area and b) mean layer thickness

The uncoated area is on one hand related to the limited width of the slot-die coating head (5-10 % are generally uncoated) and on the other hand related to coating defects and process disturbance. Also some completely uncoated samples are found. The mean layer thickness also varies significantly during the process. This indicates further influences on the slot-die process. In general, the soft modelling enables the HSI for the use as an inline monitoring tool.

Besides the given example it also possible to calculate the refraction index or the absorption of thin films. Further work was done evaluate also the sheet resistance and conductivity for conductive thin films.⁵ As it is shown in Figure 4, the sheet resistance can be determined in a non-contact way by hyperspectral imaging. In contrast to other resistance measurements (eddy current or four-point-measurement), the HSI is able to give a full resolution for the probed surface with the visualization of all defects, scratches and further contaminations.

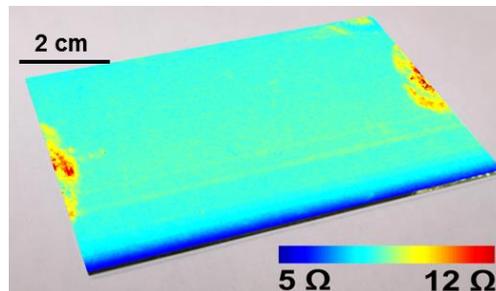


Figure 4 Sheet resistance map of transparent conductive oxide (indium tin oxide)

The decreasing resistance at the edge of the glass substrate is genuine, whereas the higher resistance values on the narrow sides did not correspond to optical model. The error map reveals high deviations for this sample area (data not shown, in this example finger prints on the surface affects this result).

If the presented concepts are thought one step ahead, nearly all critical thin film and substrate features have an influence on the spectral characteristics, i.e. stack composition, inclusions, cracks and scratches, pin holes, substrate defects, contaminations, material and gradients. The occurrence as well as the spatial distribution and frequency of all defects are hidden in the hyperspectral data. When the data evaluation is combined in a gentle way with the well-known characteristic of the sample, unusual results can be obtained by hyperspectral imaging. In this way it is possible to derive the water vapour transmission rate (WVTR) of a high barrier foil by hyperspectral imaging.⁶ This soft modelling is orders of magnitude faster than conventional methods for the determination of the WVTR, the HSI result is calculated in seconds instead of several hundred hours.

A further example for the use of soft modelling approaches is the prediction of the adhesion strength of bonded mechanical components. Normally a wedge test determines the adhesion strength within several weeks. Again the HSI is capable to do the same within seconds.⁷

These 'feature extraction applications' can be widened furthermore towards laser scribing process (selective removal), defect and contamination screening etc.^{8,9}

The Fraunhofer IWS has developed the software package **imanto**[®] *pro* to deal with hyperspectral data. All steps, starting from hardware control and data recording, as well as data pre-treatment and evaluation (also with process interface) can be performed in this software suite.

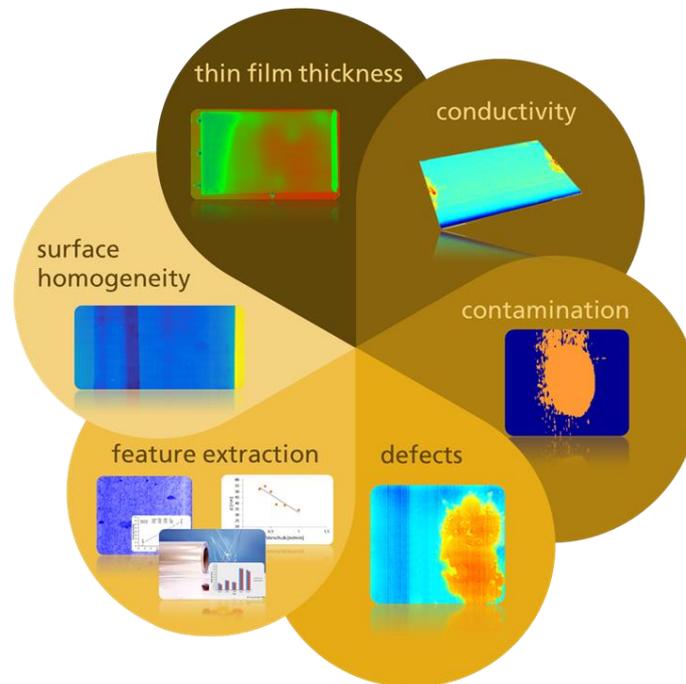


Figure 5 Summary of possible applications of Hyperspectral Imaging

In Summary, the HSI is one of the most promising monitoring technologies for webs regarding a full surface and 100 % screening. The high dimensionality of the data made the extraction of unusual features possible. Nearly all substrates, surfaces and thin film materials are available for this kind of spectroscopic imaging.

¹ Fujiwara, H.; *Spectroscopic ellipsometry: principles and applications.*; John Wiley & Sons., 2007.

² P. Wollmann, F. Gruber, W. Grählert; *Anordnung zur Bestimmung von Eigenschaften und/oder Parametern einer Probe und/oder mindestens einer auf einer Oberfläche einer Probe ausgebildeten Schicht*; patent pending, 2014.

³ F. Gruber, P. Wollmann, B. Schumm, W. Grählert, S. Kaskel; *Quality control of Slot-Die Coated Aluminum Oxide Layers for Battery Applications Using Hyperspectral Imaging.*; *J. Imaging*, 2 (12), 2016

⁴ Kessler, W. *Multivariate Datenanalyse: für die Pharma, Bio-und Prozessanalytik.*; John Wiley & Sons., 2007.

⁵ E. Weißenborn, P. Wollmann, W. Grählert; *Anordnung zur orts aufgelösten Bestimmung des spezifischen elektrischen Widerstands und/oder der spezifischen elektrischen Leitfähigkeit von Proben*; patent pending, 2015.

⁶ F. Gruber, P. Wollmann, W. Grählert; *Anordnung zur Bestimmung der Permeationsrate einer Probe*; patent pending, 2015.

⁷ P. Wollmann, F. Gruber, W. Grählert; *Anordnung zur Bestimmung der erreichbaren Haftfestigkeit vor Ausbildung einer stoffschlüssigen Verbindung an einer Oberfläche eines Fügepartners*; patent pending, 2015

⁸ W. Grählert, P. Wollmann, F. Gruber; *Anordnung zur Bestimmung der Oberflächenbeschaffenheit von Bauteiloberflächen*; patent pending, 2015.

⁹ P. Wollmann, F. Gruber, W. Grählert; *Anordnung zur Bestimmung der Tiefe von in Oberflächen eines Substrates, auf dem mindestens eine Schicht aus einem vom Substratmaterial abweichenden Material ausgebildet ist, ausgebildeten Vertiefungen*; patent pending, 2015.