From Color to Chemometrics: Strategies to determine coating thickness and quality

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Abstract

Inline and in-vacuum process control for thin-film analysis during production is an important factor to improve and stabilize product quality and reduce machine set-up and down times. ZEISS is capable to provide a variety of instruments and methods to obtain relevant process indicators.

1. Introduction

Within the glass processing and converting industry a well-known method to perform process control is the determination of color values (e.g. L*a*b*). Therefore, most coating plants for architectural glass or packaging foils are equipped with visible wavelength range inline or in-vacuum spectrometers, respectively, for two purposes: (1) Monitoring of the intended final quality of products in which color appearance is an essential feature. (2) Monitoring of color values and their variation as very sensitive indicators for the applied layer thicknesses.

However, for more complex layer designs driven by customer demands, e.g. triple silver coatings, color variations might no longer be suitable layer thickness indicators. Another method for the determination of layer thicknesses is needed. In these cases, physical method based calculation processes can be used to determine the layer thicknesses from the measured spectral data. We use FFT algorithms and model based layer analysis to obtain this information.

An additional field of application is wet coating on diffuse materials like paper. Here, too, a demand for process control and thus process-capable metrology arises. Neither color measurement nor thickness determination based upon interference are sufficient to provide stable process metrology indicators. Therefore, a method based on chemometric models was introduced to provide the machine operator with reliable process information.

2. Experimental

2.1 Example of Color as a Coating Thickness Indicator

For simple layer designs a correlation of the thickness and a color value may exist and this correlation needs to be found. This theoretical calculation can be performed prior to any real measurement. The results of these calculations for ZnO are shown in Fig.1.

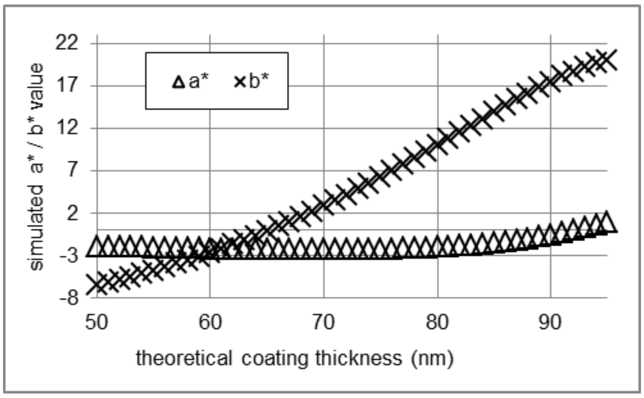

Fig. 1 Correlation of a* / b* and coating thickness of ZnO calculated with the SCOUT modelling software.

The data show a good correlation between thickness and the b* value for this specific layer.

For process control a 3rd order polynomial based on the data in Fig. 1 was used to calculate the coating thicknesses from the measured b* values.

On six ZnO coated glass panes (S1 – S6) the b* values were measured and the predicted thickness results are shown in Fig. 2 for three of the samples:

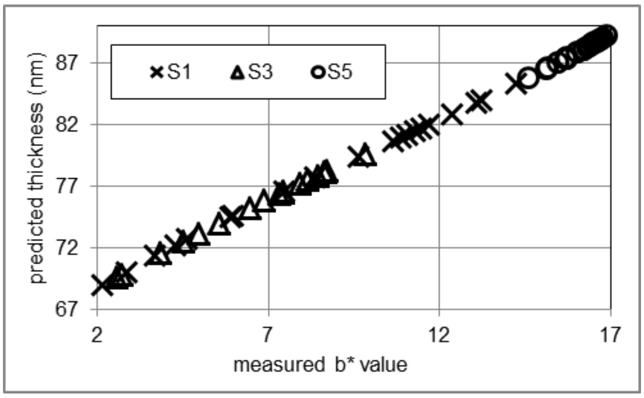

Fig. 2 Thickness results based on b* measurements

2.2 Determination of Grammage on Paper (Thickness)

The paper samples were measured on a 45°:0° diffuse reflectance probe connected to a NIR spectrometer with a spectral range of 1340 – 2000 nm. The setup was calibrated with a diffuse reference material. The paper samples' grammage values ranged from 0.19 to 4.14 g/m². Each of the 13 samples was measured at three spots. The spectra of the samples with a grammage of 0.19, 1.80, and 4.14 g/m² were used to develop a calibration function (see Fig. 3). All other measurements were used for validation of the calibration function.

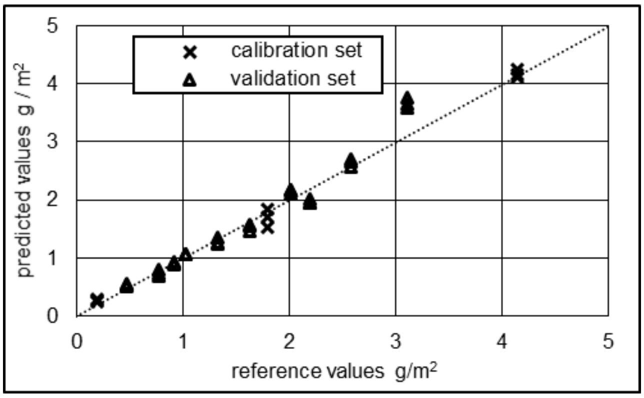

Fig. 3 Plot of the predicted grammage values for three calibration samples and 10 validation samples

3. Results and Discussion

3.1 Color and Thickness

Color determination in a process environment is not an easy task, however, color values are a stable process control indicator. If it is possible to find a correlation between color and thickness the resulting values are more stable than directly measured thickness values in terms of drift and noise.

The method was used on a standard traverse measuring system provided with two spectral reflection (for substrate and film side) and a transmission measurement probes.

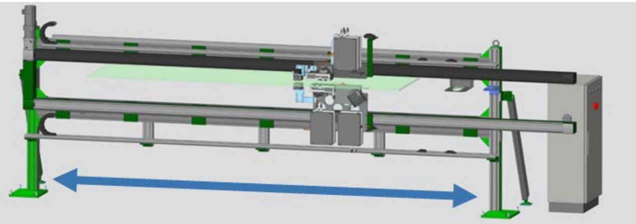

Fig. 4 Traversing measurment system, the arrow indicates the moving range of the measurement probes

The example shown in Fig. 5 demonstrates the tuning of the coating process to bring the layer to specified thickness.

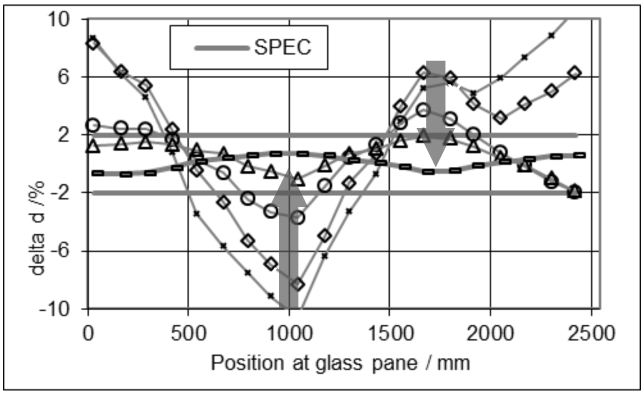

Fig. 5 Example of a cross section plots of coating thickness variation (d/% of nominal value) for process monitoring (six different glass panes)

3.2 Chemometric Prediction

The absorption peaks of the coating material in the spectral range between 1600 and 1800 nm can be used to quantify the grammage of the coating. The mean deviation between the predicted and the reference grammage values was 0.09 g/m². For the sample with the reference value of 3.11 g/m², a grammage of 3.6 g/m² was predicted. The outlier is currently under investigation.

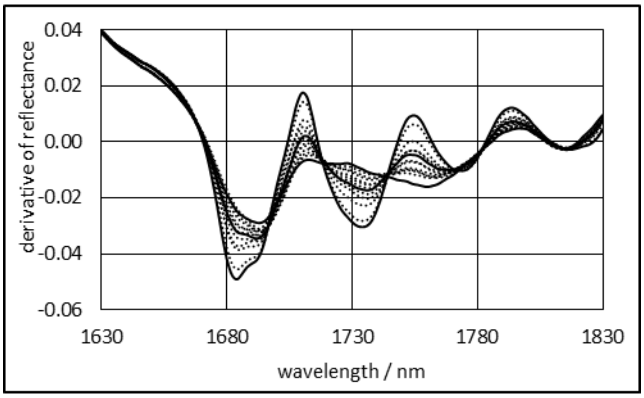

Fig. 6 First derivative of the reflectance spectra. The solid line spectra represent the calibration set; the dotted line spectra represent the validation set.

3.3 Complete Quality Control

The final product quality is defined as a combination of many features like color appearance or coating thickness. Since inline spectrometers measure the full spectrum these quality-related parameters can be evaluated in one single step.

Broadband inline spectrometer systems allow chemometric prediction in the NIR range as well as color evaluation, even within a single measurement system.

4. The Importance of Accuracy

4.1 Color Measurements

If color appearance itself is a quality criterion it can be measured directly. Nevertheless, a ISO / CIE standardized setup for accurate measurements (comparable with standards, inter-instrument agreement) is mandatory. This includes a full spectrum sensor requested to overcome metamerism issues [1].

4.2 Different Methods Accuracy Needs

Needless to say that accuracy in terms of tractability to standards is a must for quality assurance tasks. For inline process control it seems sometime sufficient just to use precise, in terms of reproducibility, instruments and methods.

- *Color as thickness indicator*: Photometric accuracy is important for this method cause the color is the thickness indicator. The pre-measure prediction is based on an ideal instrument, so the instrument in use need to be nearly ideal.

- *Chemometric prediction*: Will work with non-accurate (but precise) systems as well.
BUT: The developed methods will only work on the system they are developed with
    - No scaling possible
    - In case an instrument or parts of the system needs to be changed, the method needs to be adopted
- *Peak method*: Only wavelength accuracy needed cause the position of the peak is related to the coating thickness
- *White light interference*: Only wavelength pitch accuracy needed cause the frequency of the interference pattern is related to the thickness

4.3 Accurate Instruments

It was shown, that at the discussed methods need accurate instruments.

The standard method to calibrate an inline capable spectrometer for reflection measuremts is the use of a reflection standard (mirror or diffuse white standard). Methods for absolute calibration of laboratory spectral photometers exists [2]. First designs of inline capable measurement absolute probes are available [3].

Fig. 7 Inline capable measurement probe for absolute spectral reflection and transmission results

5. Conclusion

It is shown that inline spectrometers are versatile and stable process-capable measurement systems to keep a production line stable and ensure the final product quality [4].

The data provided by such instruments can be used for direct monitoring of the production process. Because the quality of the measurement results is very stable and data collection is fast, they can be used as an input for automated control loops. However, this needs a close cooperation between process engineers, automation solution provider, and measurement system provider.

With the introduction of different methods to obtain coating thickness from inline spectra data there is a bigger freedom of choice between methods. This will help to introduce inline spectroscopy in fields, where more complex methods, e.g. XRF, are in use these days.

Acknowledgements

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