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Summary

This summary of PRODI project deliverable D1.4 provides an overview of the work done in the Work Package one (WP1) to define roll-to-roll (R2R) requirements for measurement instruments and automation systems. Analogous to the previous deliverable report, the definition of roll-to-roll (R2R) manufacturing equipment requirements was focused on three end applications including: organic solar cells (OSCs), electrochromic displays (ECDs) and organic thin-film transistors (OTFTs).

First, the relevant parameters for measurement and automation were identified from the previous study (D1.2) on the requirements for manufacturing equipment. These parameters are discussed in Chapter 2. From these parameters the following main parameters for the abovementioned applications were identified:

- Overlay and registration,
- Layer uniformity,
- Pattern quality,
- Product functionality.

The requirements for measurement and automation solutions for these parameters are quantified and, if not quantifiable, discussed in Chapter 3. Chapter 4 shows an overview of the currently available measurement and automation solutions to ensure that the requirements for these parameters are met. In the final chapter, the conclusions of this research are summarized.

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1 Background & objectives

Within the new emerging technology of printed and large area electronics, a huge number of applications have been proposed and an evolution of the complexity of devices and systems are foreseen. This expectation set forth new requirements on low-cost production and control systems in order to preserve high quality along with rational production routes. The aim is to use the technical and application requirements on production equipment as a basis to derive essential requirements for respective measurements, control and automation.

The main emphasis will be on the product characterization measurements and process control in the R2R polymer and printed electronics industry. The contributors do have also a lot of expertise on various materials (both organic and inorganic) and other aspects of the manufacturing, and that background should be used in completing the study.

The first thing in collecting the requirements is to select appropriate amount of applications to focus on, and define the set of parameters and characteristics to be collected as industrial requirements. The actual contacts with the industrial representatives are essential to form an accurate picture of the situation. The expertise of the Industrial Advisory Board will be needed to successfully complete this task.

The key objectives include defining the state-of-the-art in R2R measurement and automation, collecting and formulating requirements for the future, and contributing to the roadmap for the European R2R polymer and printed electronics machinery and automation industry. The practical goals include:

- Setting the necessary guidelines, with which the European R2R measurement and automation suppliers can determine the amount of work needed to enter the organic R2R manufacturing segment, and the most promising technology choices for the R&D
- Supporting the measurement and automation companies in their strategic decision making regarding the markets, technologies, applications, and roadblocks

The target is to find specific, distinguishing requirements for the measurement instruments and automation systems to be used in R2R electronics manufacturing processes. The applications analysed in the manufacturing section should be used also in this work. For each application a set of characteristic parameters are being defined in order to form a solid base for the requirements gathering.

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2 Relevant parameters for measurement and automation

2.1 Ink properties

2.1.1 Viscosity

Viscosity is the measure of the internal friction of ink, thus describing the flow behaviour of the ink during the printing process. The viscosity depends on the shear rate and temperature: the ink viscosity varies during the printing process since ink encounters high shear rates in ink transfer points and the process temperature tends to change to some extent during a single print run. In addition, the viscosity decreases with increasing process speed because of increased shear. The ink viscosity should be adjusted and optimized according to the desired printing method since too low viscosity leads to excessive ink spreading and too high to difficulties in the ink transfer.

The ink viscosity should be controlled periodically/continuously during the printing process to ensure a stable ink viscosity and consistent printability. Efflux cups are used to measure periodically the static viscosity of the ink whereas viscometers can determine the static or dynamic viscosity. Both of these methods can be used in-line in ink tanks but they do not tell anything about the ink viscosity in the printing process. Rheometers, on the other hand, can simulate the dynamic conditions of the printing process reliably but the measurement is always done off-line.

The desired ink viscosity in the printed electronics applications depends on the deposition method, required ink layer properties, and device properties. For example, gravure printing requires lower viscosity than screen printing, and thinner layers are obtained with lower viscosity inks. The adjustment of the ink viscosity is often done by changing the solvent amount but the material solubility limits the achievable viscosity range.

Organic materials used in printed electronics applications have rather poor solubility, thus limiting the ink viscosity. This can lead to poor device performance because of the difficult control of the printing process and uneven printability. Therefore, the trend is towards higher viscosities. Inorganic active materials are typically dispersed into the ink solvent as individual particles. There are several commercially available metal inks for all the main printing methods that come in different viscosities and solids contents. However, other inorganic inks have difficulties in producing good coverage and high performance at low viscosities. Therefore, higher viscosities are needed. The target viscosity of organic and inorganic active inks is shown in Table 1.

Table 1 Typical and target values for viscosity of both organic and inorganic active inks.

Viscosity [mPas]	Value	Target
Organic inks	<30	>50
Inorganic inks	>5	>200

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2.1.2 Surface tension

The surface tension of the ink describes the wetting and spreading behaviour of the ink on the surface of the substrate. In addition, the surface tension plays a major role in the ink adhesion. The surface tension of the ink should be lower than the surface energy of the substrate/previously deposited layer to allow proper ink layer formation onto the substrate.

Due to the fact that printing processes are fast, the measurement of dynamic surface tension is desirable. However, there are only off-line measurement methods that can simulate the dynamic conditions encountered during printing. These methods include maximum bubble pressure, bubble formation rate, drop volume, and dynamic contact angle measurements.

The surface tension requirement of the active inks depends on the surface energy of the substrate or previously deposited layer in order to obtain good print quality and proper adhesion. Currently, the surface tension of the inks lies between 25-70 mN/m but there is a trend towards lower surface tensions to allow trouble-free deposition on the substrate with rather low surface energy. In addition, the upper the ink layer, the lower the surface tension. However, the ink wetting ability can be improved by pre-treating the substrate/ink layers to increase their surface energy.

2.1.3 Solid content

Solid content represents the amount of solid material in the ink. The solid content of the ink affects the ink layer thickness, the flow properties and printability of the ink, and the layer performance/functionality. The higher the solids content, the more active material is deposited onto the substrate, thus increasing the layer functionality. However, at the same time, the ink transparency suffers.

Solid content measurements are typically made off-line. The simplest method is based on weighing and solvent evaporation but there are also methods that are based on changes in for example ultrasound, acoustic wave, or microwave signals. Some of these methods can also be applied into the actual printing process.

In printed electronics applications, the solids content of the ink depends on the material solubility and printing method. The trend is towards higher solid contents since the solid content of organic active inks is currently below 3 wt-%. The solid content of inorganic inks is 10-90 wt-% but the trend is to keep the solid content between 50-70 wt-% without affecting the printability and performance. The typical solid content values and targeted values are presented in Table 2. However, too high solids content leads to printability and ink application problems. In addition, the poor solubility of the active materials decreases the obtainable solids content.

Table 2 Typical solid content values and their target values.

Solid content [wt-%]	Value	Target
Organic inks	<3	>5 (>10)
Inorganic inks	10-90	50-70

2.1.4 Drying properties/system

The ink layer should be completely dry when entering following deposition/converting stages to avoid ink sticking and poor layer quality. In addition, the ink layer should

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withstand any chemical or physical strains encountered during the manufacturing process without sticking, smudging, cracking, peeling, softening, or losing coverage. The most common drying properties are ink adhesion, solvent/other residues, blocking (sticking in stacks or in reels), and chemical compatibility.

The ink drying properties are measured off-line from a dried sample. The most common measurements techniques include determination of residues, rubbing, folding, stacking, scratching, compressing, and elongating. In addition, chemical changes, such as the amount of bonding or degree of cross-linkage, can be measured.

The drying properties of the active inks affect the printability and runnability as well as the device quality and performance. Poor adhesion leads to print defects that lead to poor device performance. Solvent or other residues increase the risk of smudging, sticking, and dissolution of other active layers. It is therefore preferred to increase the adhesion of the ink layers and ensure the layer dryness. Some active layers may also be sintered to obtain uniform ink films. If the layer is poorly sintered, the device has poor performance.

2.2 Substrate properties

2.2.1 O_2 and H_2O Permeation

Water vapour transmission rate (WVTR) is a steady-state rate at which water vapour permeates through a substrate at specified ambient conditions, i.e., ambient temperature and relative humidity. The ambient conditions together with the substrate thickness affect the WVTR value. Oxygen transmission rate (OTR) is the amount of oxygen permeated through the substrate at given conditions over a given time period. Any pinholes will increase both WVTR and OTR values. The WVTR and OTR unit is typically $g/m^2/day$ and $cm^3/m^2/day$, respectively.

The most common WVTR and OTR measurement methods include calcium test and the use of infrared or coulometric sensors. In the calcium test, a layer of calcium is patterned onto a transparent substrate. This layer is covered with a transparent barrier. Water and oxygen permeation through the substrate into the calcium layer converts Ca into transparent salt, thus changing the optical transmission of the layer that can be measured. The detection limit of the calcium test is in the order of $10^{-6} g/m^2/day$. However, the calcium test is more sensitive to water vapour than oxygen, thus making it more appropriate method for WVTR values. Infrared or coulometric sensors have a detection limit of $5 \cdot 10^{-4} g/m^2/day$. These equipments have two chambers that are separated by the substrate to be measured. The other chamber is flushed with nitrogen and the other one with pure oxygen or air saturated with water vapour. The partial pressure difference between these chambers creates a driving force for the water vapour or oxygen to permeate through the substrate. An infrared or coulometric detector is then used to detect the amount of vapour/gas permeating through the substrate. WVTR and OTR values are recorded after a steady-state has been reached or as a function of time. The main disadvantage is the long time to measure WVTR and OTR values.

In printed electronics applications, water and oxygen permeation into the active layers of the device decrease the lifetime significantly. The permeation requirements will depend on the sensitivity of the used organic materials to O_2 and H_2O . WVTR and OTR

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values are typically measured from both the substrate and barrier layers. In order to achieve long lifetimes, WVTR values should be $<10^{-5}$ - 10^{-6} g/m²/day and OTR $<10^{-3}$ cm³/m²/day. These values are significantly lower than it is possible using any plastic substrate and flexible barrier layers in use today. Further developments of the plastic and barrier materials as well as deposition process (avoid pinholes) are therefore required to increase the WVTR and OTR values. Currently, the maximum WVTR value obtained for a barrier layer is 10⁻⁵ g/m²/day. The general requirements for O₂ and H₂O permeation are presented in Table 3.

Table 3 Target values for WVTR and OTR in printed electronics applications.

Parameter [wt-%]	Value	Target
WVTR [g/m ² /day]	$> 10^{-3}$	$< 10^{-5}$ - 10^{-6}
OTR [cm ³ /m ² /day/atm]	$> 10^{-3}$	$< 10^{-3}$

2.2.2 Surface energy

The surface energy of the substrate determines the wetting behaviour of printing ink on its surface. The surface energy should be higher than the surface tension of the ink to obtain proper ink wetting and spreading as well as adhesion. The surface energy is often increased by plasma or corona treatment or chemical etching.

There are no direct measurement methods for the surface energy of a solid surface. Typically, liquids with known surface tensions are placed on the substrate and their contact angles are recorded with goniometry. The surface energy is then calculated from the obtained data.

The surface energy of plastic substrates is typically between 20 and 50 mN/m. In order to achieve proper ink wetting, the surface energy should be increased from these initial values. Typically, the substrate is pre-treated (plasma or corona treatment) to increase the surface energy. The trend is also to manufacture plastic substrate with higher surface energy in order to reduce the need for pre-treatments, thus simplifying the manufacturing process. The future targets for the surface energy of the substrate are shown in Table 4. The surface energy of the deposited active layers should be higher than the surface tension of the ink used to print the following layer. This requires ink development and/or pre-treatment steps.

Table 4 Future targets of surface energy.

Parameter	Value	Target
Surface energy [mN/m]	20-50	>50

2.2.3 Surface roughness

Surface roughness describes the layer topography as well as irregularities. The smoother the ink layer, the more even and uniform ink layers can be deposited onto its surface. In addition, the risk for ink layer roughness and poor coverage decreases.

Surface roughness can be measured using either contact or non-contact methods. In contact methods, a stylus is dragged along the sample surface in order to obtain the surface profile. Profilometers are typically used. Non-contact methods include for instance interferometers or confocal and electron microscopes. The measurement devices give information about the vertical deviations of the surface of the sample.

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In printed electronics applications, the surface roughness of the substrate and printed layers should be as small as possible to obtain homogeneous and even ink layers with good coverage and without pinholes or cracks. The ink gets evenly into intimate contact with the substrate if it is smooth, thus forming good print quality and proper device performance. Table 5 presents the target values of the substrate and ink layer roughness.

Table 5 Target values of the substrate and ink layer roughness

Parameter	Value	Target
Roughness [nm]	< 50	< 10
Layer roughness [nm]	< 100	< 5

2.2.4 Transmittance

Transmittance of the substrate describes the amount of incident light at a specific wavelength that passes through the sample. This factor is also related to the transparency of the substrate: the higher the transmittance, the higher the transparency.

Transmittance can be measured with spectrophotometers or spectrometers at a certain wavelength range. There are equipments that operate in the UV region and some operating in a wider wavelength scale. The transmittance can be measured for both substrates and printed layers. In the case of printed layers, the transmittance of the substrate is subtracted from the measured transmittance of the ink layer printed onto the substrate.

The substrate should be as transparent as possible to enable unrestricted light travel into the electronic component. For example, the high transparency of the substrate ensures that most of the light reaches or exits the active parts of the printed electronic component. There is a trend towards higher transmission values so that light can travel more easily without scattering or absorbance. Some printed layers might also have transmittance requirements that are similar to those of the substrate. Table 6 shows the target values for the substrate and some printed layers.

Table 6 Target value for substrate and ink layer transmission.

Parameter	Value	Target
Transmission [%]	> 80	> 90

2.2.5 Registration

Print register is the positional accuracy of the overprinted ink layers. In multilayer printing, it is essential to differentiate between circumferential, lateral, and diagonal register. The register accuracy in overprinting lies typically within a range of a few tens of nanometres. The main parameters affecting the register accuracy are the dimensional stability of the substrate and ink layers and manufacturing process instabilities and parameters.

Register systems can be installed in-line and some of the systems can adjust the register automatically. Register control systems use opto-electronic sensors to detect register deviations on register marks that are printed onto the substrate by each printing unit onto non-image areas. The size of the register marks is typically 1-1.5 mm and the

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measurement accuracy can be as good as 5 μm . Figure 1 illustrates an example of an register mark and register error.

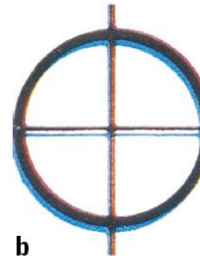


Figure 1 Register mark with a register error in overprinting. [Kipphan, H., Handbook of Print Media]

In printed electronics applications, the register accuracy should be as good as possible between active layers to achieve functional devices. Currently, the register accuracy is better than $\pm 50\text{-}100 \mu\text{m}$ in overprinting in R2R processes but the trend is to improve the accuracy significantly. The target value for registration accuracy in printed electronics applications is shown in Table 7. The dimensional stability of the substrate and ink layers as well as the process instabilities and parameters have a significant effect on the registration. The main problem lies in the elongation and shrinkage of the flexible substrate. The register accuracy requirement depends on the application.

Table 7 Register accuracy targets.

Parameter	Value	Target
Registration accuracy [μm]	$< \pm 50\text{-}100$	$< \pm 5$

2.3 Substrate handling parameters

Substrates are affected by the way they are handled; handling both before the substrates are being used as well as in the actual printing process. Handling involves general environmental parameters like the storing and production ambient but also more specific parameters like how the substrate is mechanically affected when run through the system. An example of the latter is the speed of the substrate through the printing process and the web tension. The amount of stress applied to the substrate is proportional to the speed. Ambient parameters are discussed more in detail in the overall process paragraph. Other parameter important for the handling of substrates not discussed elsewhere is web cleanliness and Corona treatment.

2.3.1 Web cleanliness

The web can be cleaned by various different methods like oscillating web cleaners, brushing and/or adhesive rollers. The nip roller arrangement using adhesive is most effective in penetrating the thin film of air attached to all moving webs known as boundary-layer air. The expected range of requirements on speed and web cleanliness parameters is shown in Table 8.

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Table 8 Expected range of requirements for substrate handling.

Parameter	Short term	Medium term	Long term
Speed (m/min)	5	50	300
Web cleanliness (particles/cm ²)	100 < 50µm	50 < 10µm	10 < 2 µm

2.3.2 *Web corona treatment*

To be able to print on for example plastic that has long molecule chains the surface energy must be higher on the substrate than that of the ink used. To obtain a higher surface energy on the substrate that shall be printed or coated on Corona treatment is a suitable method. Corona is a high frequency electric discharge towards the surface of the substrate, the result of this is that the long molecule chains are broken up and a high number of valences are created. The ozone from the electric discharge also creates carbonyl group that increases the surface energy. It is preferred to use foils that are treated at manufacturing since it is harder to treat foils that have been stored without having been initially treated. The Corona treatment should be performed in line just before the printing or coating. The unit for measuring surface energy within the printing industry is Dyn/cm, equivalent to mN/m. A water based ink holds about 48 Dyn/cm or 48 mN/m.

Commonly used plastic films are:

- PET (Polyethylene terephthalate); used for printing, high tensile strength
- PE-LD, PE-HD (Polyethylene); often used for coating of paper substrates to make a dense printable surface
- PP, BOPP (Polypropylene); often used as encapsulation and for adhesive tapes

Material	mN/m	Process/ink	mN/m
PP	29	Solvent based inks	40-42
PE-LD	31	Water based inks	46-48
PE-HD	32	Coating	44-54
BOPP	32	Lamination	46-56

2.4 **Patterning/Coating/Lamination process parameters**

2.4.1 *Web tension*

Due to the web tension the substrate can be elongated in the machine direction (MD) when passing through the printing equipment. Also the heat from drying or curing stations can affect the dimensions of the web, in this case also in the Trans machine direction (TD). A typical value for thermal shrinkage is 1.0 % in both MD and TD for PET foil with a thickness between 50 to 250 µm exposed to 150 °C for 15 minutes.

The elongation in MD due to web tension should be controlled by keeping the web tension to a minimum still enough to obtain control over the web. If there is still a elongation at the minimum possible web tension this can be compensated for when designing the print forms or in the data files when using digital printing. There is also in some equipment possible to continuously compensate for size variations (as well as

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registration) by letting the print form move with a slightly different speed than the web using printed register marks as steering guides.

As printed electronics strives to use as thin substrates as possible, and thereby sensitive to mechanical and thermal stress, focus must be on controlling web tension, curing temperatures and times and the use of advanced equipment to measure and compensate for size variations

2.4.2 Web positioning

Positioning is another important for patterning processes. The parameters affecting the registration accuracy are X/Y positioning and angular positioning of the printed image in each printing step in relation to the registration marks or the previous printed image. Digital printing or computer to print form (without film masks etc) is preferable. Sleeves for Flexo and digital engraved screens give a higher precision and less angular deviation and also allows for seamless continuous patterns if needed.

If the substrate dimensions are changed during the transport through the machine due to mechanical tension temperature or air humidity it have to be compensated for when designing the different print forms or digital files for the different printed layers.

The Z positioning is depending on the layer thickness on the previously printed layer. If a layer is to be printed over the edge of a previous layer (for interconnect) a high step creates problems for producing a continuous film without an electric break or high resistance over the break. Therefore thin layers are preferable.

Table 9 Expected range of requirements for process parameters.

Parameter	Short term	Medium term	Long term
X, Y-position (registration)	≤ 50µm	≤ 25 µm	≤ 5 µm
Z-position (leveling), layer thickness, step coverage	≤ 40 µm	≤ 20 µm	≤ 10 µm
Orientation (angular positioning) max deviation	≤1.5°	≤ 1°	≤ 0.1°

2.4.3 Layer uniformity

Spectroscopic / false-colour measurement

Coverage and uniformity measurement can be done with standard machine vision camera either by transmission or reflectance measurement. With this kind of measurement is it possible to enhance discrimination by selecting wavelength to absorption of the actual layer (Figure 2). Even without any computational analysis, this kind off picture reveals the imperfections in the film’s uniformity. Images can then be analysed for fill factor calculation, pin-holes, etc.

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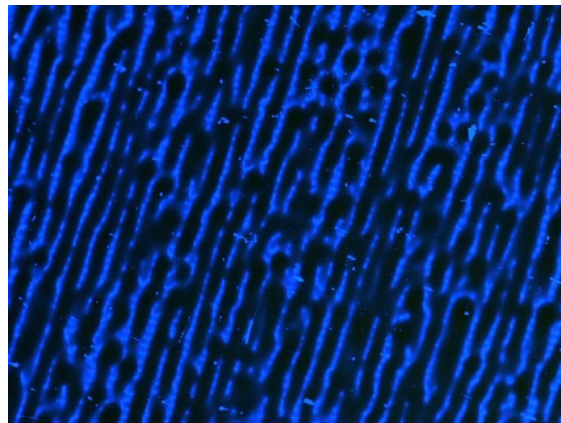


Figure 2 Uneven thin film printing quality with transmission measurement.

Thickness is in principle possible to measure with the spectroscopic measurement, but interference and multilayer structure will disturb measurement. In some cases, interference can be voided by selecting measurement wavelengths from area where is no interference. With multilayer one idea is to select point where is only one layer from layout (this can be a test printing square in the side of layout).

Interference measurement

Interference measurement is typical approach to thin film measurement and it has been widely used with plastic films. This method can also be used to printed electronics thin films and it can give average film thickness with good accuracy. By compounding average thickness information with previous kind false-colour measurement (picture 1) quite good idea of the quality can be achieved. Interference measurement's weak point is that you have to know optical parameters (refractive index) of the materials and in multilayer structure case mathematic can be very complex.

Ellipsometry

Ellipsometry measures the change of polarization upon reflection or transmission and with it is even possible to measure thin films that are thinner than the wavelength of the measurement light. As a restriction thin film layers must be well-defined and optically homogenous and isotropic and at the same time number of the layers must be small. Method also need's experimental parameters and math is very complex, so this method is not usable in printed electronics pilot trial where materials change continuously.

2.4.4 Pattern quality control

Pattern quality is a difficult parameter to quantify. In principle, the pattern should match the designed pattern in terms of scale, shape, completeness, contrast, etcetera. Generally, template matching and registration techniques are used to detect differences between the applied and designed patterns and quality is evaluated based on specific features in the observed differences. It is expected that the design of the pattern will also be based on the expected quality of the patterning process and that the resulting quality control features are therefore specific for a certain given combination of design and manufacturing technology.

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2.5 Overall process

2.5.1 Ambient conditions

For production process of for Polymer and Printed Electronics it is needed to control temperature, humidity; gas composition of the air in the environment of the production facilities and storage. For safety reasons the measurement of solvent concentration can be important.

Flexible substrates for flexible electronics tend to deform as a function of temperature and humidity. For example, Polyimide (PI) foil can absorb 1.8 % of water, which can result in an expansion of about 0.1-0.2 %. For some other foils like Polyethylene Terephthalate, the absorption is about 0.14 % which results in an expansion of 0.025%. For process control and for patterned techniques, it is important to control the dimensional stability of the substrates to meet the overlay requirements of the different layers.

The process conditions are important for the coatings that will be used in the process. In some cases production in enclosed environment with oxygen less, low humidity or even vacuum can be needed, which requires the use of gloveboxes, or closed systems. Change in temperature and humidity can cause dew on surfaces and change properties of functionalized layers.

Other reason for controlling the humidity is the static electricity. At a low humidity, the charging of surfaces is higher.

The relative humidity in air depends on the amount of water vapour, temperature and pressure. Assume air at 21 °C, with a relative humidity of 50%, then the relative humidity will rise to 100% at 10 °C which means condensation (see Figure 3).

The relative humidity can be calculated with:

$$\%RH_{(T)} = \frac{(\%RH)_{ref} \cdot (P_w)_{T.ref}}{(P_w)_T}$$

Where $\%RH_{(T)}$ [-] is the percentage relative humidity at any other temperature, $(\%RH)_{ref}$ [-] the reference percentage relative humidity of the air sample, $(P_w)_{T.ref}$ [Pa] the vapor pressure of water of the reference air sample, and $(P_w)_T$ [Pa] the vapor pressure of water at any other temperature. These values of the vapor pressure of water can be found in books of constants. For example when air at 21 °C and 50 %RH is cooled down to 15 °C without changing the ambient pressure or the water contentm the humidity will increase to 73 %RH.

$$\%RH = \frac{\%RH(21^{\circ}C) \cdot P_w(21^{\circ}C)}{P_w(15^{\circ}C)} = \frac{50 \times 2486}{1704} = 73\%$$

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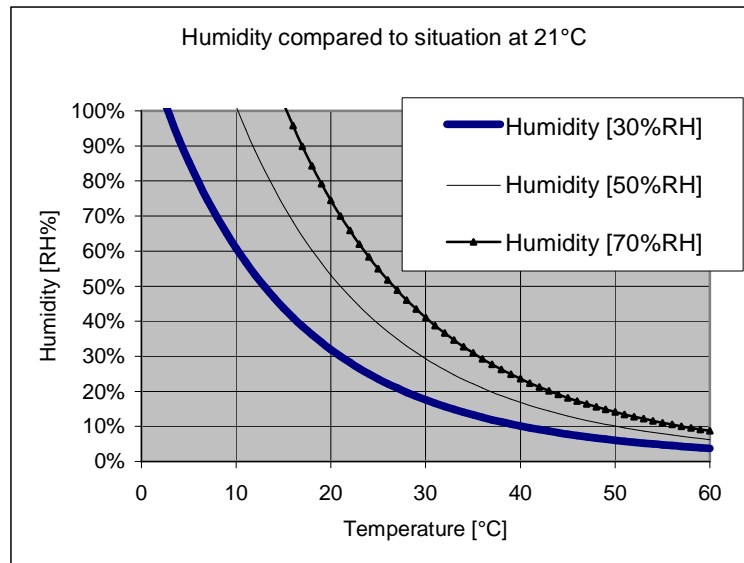


Figure 3 Humidity as a function of temperature at a constant water vapor in air.

Measurement equipment for measurement of temperature and humidity different types of thermometers are available. For humidity, an overview is given in Section 4.3.2. The methods described there also apply to the ambient measurements.

Solvent concentrations must be controlled in the production machines. In case of exceeding the safety values, an alarm should be sounded to inform people, the production process (*e.g.* coating or drying) should be aborted, and the air ventilation should be increased. The type of the detection system depends on the used solvents.

2.5.2 Process monitoring and control

The ultimate goal of implementing process monitoring and control is yield optimization. Typically, a computer integrated manufacturing (CIM) system used in silicon semiconductor fabrication plants generally consists of a manufacturing execution system (MES), process equipment interface (PEI), an automated material handling system (AMHS), and an engineering analysis system (EAS)². Part of the EAS, the yield and device performance are analyzed and optimized using advanced process control (APC). APC is an approach to process control to either correct the course of the manufacturing process or to manage faults in the produced products using various kinds of process control tools⁴, such as statistical process control (SPC), run-to-run (R2R) control, and fault detection and classification (FDC). FDC is usually used at the equipment level large amounts of process data are analyzed to monitor equipment performance. R2R control is used within a lot or within a collection of lots to adjust processing parameters on a wafer-by-wafer basis. In this way, incoming variability as well as changes in equipment performance can be monitored. In general R2R compensation occurs over longer time periods using less data than FDC, and is focused on overall process yield issues rather than defect detection on individual products. SPC applications may control metrology operation and post process data obtained from the whole process line using statistical analysis techniques to relate the overall process parameters to yield and device performance. Since the 60's the trend has been from solely SPC towards using all methodologies such as FDC, also enabled by the advances in information technology that enable the processing of ever increasing amounts of data

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in ever decreasing amount of time. Nowadays, sophisticated real time monitoring sensor/actuator control systems with feed back can also be implemented.

Whereas the silicon based semiconductor industry is moving towards to real time process control, roll-to-roll manufacturing techniques for organic semiconductors rely on real time process control. It is clear that this is a very challenging starting point. From a control theory point of view, the continuous roll-to-roll manufacturing of electronics products opens opportunities for full feed back control. Also, recent developments in the fabrication of flat panel displays and photo voltaic panels provide opportunities in terms of the rapidly increasing capabilities of the inspection systems from numerous manufacturers (*e.g.* Veeco, KLA Tencor, Orbotech, Dalsa, *etc.*) that are able to inspect up to generation-10 sized flat panel displays (*e.g.* Sharp). These systems are in principle capable of monitoring pattern quality on a sub-micrometer scale inline and the extension to processing very large areas per unit time appears to be mainly dependent on the limits of real time data processing.

A transition from flat panel displays towards flexible flat panel displays seems straightforward and then a logical step would be using roll-to-roll technology. Because the roadmap for full organic electronics technology is competing with the existing international technology roadmap for semiconductors (ITRS), which has a strong focus on inorganic semiconductor technology based on silicon on wafer or glass, only the potential of producing low cost large area electronics, such as low-efficiency photovoltaics on foil and OLED's for lighting and signage are identified at present. This is the main driver for using roll-to-roll manufacturing techniques to produce organic electronics. For (organic) TFT's overlay accuracy and critical dimensions are vital for device performance, and the transition from foil-on-substrate manufacturing to large area roll-to-roll manufacturing seems a bridge too far at present, and an intermediate manufacturing technology such as sheet-to-sheet is foreseeable. Although the organic and silicon electronics roadmaps currently coexist, it is expected that they will coincide that at some point in the (near) future. This is already happening for flat panel displays, where OLED displays were recently introduced (*e.g.* Sony). Nevertheless, the current gap in manufacturing technology maturity is eminent and significant efforts are still required before roll-to-roll technology can be considered as a viable technology for manufacturing either organic or inorganic high performance electronics.

2.5.3 Functional inspection

Presently, functional inspection of product parameters during roll-to-roll manufacturing of organic electronics is deemed vital to ensure adequate product yield. At the same time that the control of production of silicon based products has experienced a paradigm shift from SPC towards the broader oriented APC (see previous section), a trend from measuring product states to measuring the process states is visible. While the paradigm shift can mainly be attributed to the enormous technological advances in information technology and the ever increasing demands for minimizing manufacturing costs, the shift in monitoring is also possible because of the maturity of the production methods for silicon electronics. In general, the need for functional testing during manufacturing decreases as the maturity of the product and the manufacturing process increases (see Figure 4). This is understandable because in time the relations between certain process parameters and product functionality will become apparent, *e.g.* problems with overlay (process parameter) will generally decrease organic TFT device performance (product parameter). This knowledge can be used to optimize the process or even change of the

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product design but also to replace expensive monitoring wafer methods by less expensive process monitoring methods.

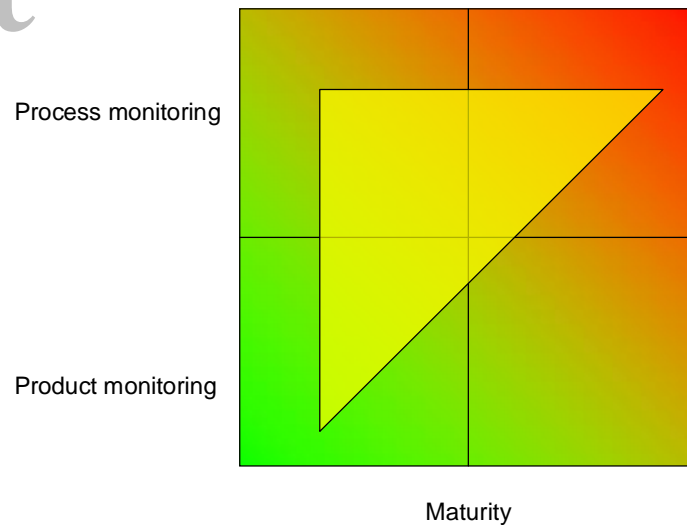


Figure 4 Monitoring focus as a function of product and process maturity.

The manufacturing of products of organic electronics with roll-to-roll technology is only starting to reach production scale maturity and still much effort is required for the development of the products and suitable production and metrology technology. This means that both product as well as manufacturing process maturity are low. Therefore, the success of the product-process combination is still very dependent on tight monitoring of both the relevant process as well as the relevant product parameters during manufacturing.

Existing metrology solutions that are used for functional testing of silicon electronics are generally not compliant with roll-to-roll manufacturing on large areas. On the other hand, metrology solutions available from the printing industry are generally not compliant with the dimensional requirements for electronics and also do not cover functional. Because of this technology gap, a trade-off between production quality and production capacity is enforced. In principle this could create an incentive to develop new methods for high volume inline testing of large area electronics, were it not for the fact that for the same reason production volumes are so limited that the economic drivers are simply hard to find. Currently, the lagging development of monitoring equipment is considered as one of the possible red brick walls for roll-to-roll manufacturing of electronics.

It is in this view that functional testing of sub assemblies and the final product should be considered here. Because the roll-to-roll manufacturing methods very often consist of a sequence of sub-processes (*e.g.* cleaning, levelling, coating, patterning, lamination) the functionality of sub-assemblies can be tested separately. Evidently, the requirements for the functional tests are dependent on the type of the sub assembly. Functional testing of the full assemblies is treated for the different applications (OTFT, EC-displays, OPV) considered in this study in Section 3.4.

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We will discern the following sub assemblies in this section:

- Barrier layers
- Electrode/conducting patterns
- Conducting patterns
- Dielectric layers
- Semiconductor layers
- Encapsulation layers

Barrier/encapsulation layers

The most important properties of the barrier/encapsulation layers are the oxygen transmission rate (OTR), water vapour transmission rate (WVTR), and thermal stability. Because the material properties are part of the design of the product and final product functionality may be affected by exposure during the production process, product functionality is typically ensured by monitoring the thickness and uniformity of the deposited layers (see Section 2.4.3).

Electrode/conducting patterns

Relevant parameters of the electrodes are the contact and sheet resistance. Both can be measured using 4-point probe measurements. Other methods are tank circuit eddy current measurements, and front side mutual induction eddy current measurements. Because eddy current techniques are contactless, such measurements are highly suitable for measuring the electrode patterns on a moving web. For conducting tracks, line resistance is the relevant parameter to test.

Dielectric layers

For the OTFT, the capacitance of the dielectric layer is an important factor determining device performance. Therefore it is essential that this feature is produced within specification. Apart from the geometric parameters (effective surface area of overlap between source, drain and the gate contacts, and the thickness) the permittivity (κ) is an important material parameter. However high- κ materials, such as Ta_2O_5 , are generally electrically incompatible with organic semiconductors and therefore organic modifier layers are frequently applied between the dielectric layer and the semiconductor layer. Any method for testing the dielectric properties should be able to cope with the combined properties of the constituting layers.

Analysis using spectroscopic ellipsometers depends on an appropriate model of the optical behaviour of all layers present. Examples of such models are tabular models, harmonic oscillator (HO) models, the Cauchy polynomials, and the Bruggeman effective medium approximations. The precision of the parameters in these models decreases significantly as the thickness of the layer decreases and the applicability of these models is dependent on the dielectric materials¹. An alternative approach could be using the measured spectral data of a well functioning dielectric layer as a template for comparison with new measurements, provided that the spectral data is to some extent uniquely definable by the desired layer structure and optical properties.

Besides ellipsometry other non-contact methods exist, such as Quantox (KLA Tencor) or SASS (SDI). These measurements also enable the measurement of the electrical properties relevant to OTFT performance and provide means to predict final device operation during the manufacturing process.

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The methods described in this section provide excellent opportunities for applications in roll-to-roll manufacturing techniques. Although the scale up of these metrology solutions will pose important technological challenges it is expected that it will be more challenging to actually deposit the layers and to ensure proper deposition of the electrodes than to monitor the resulting device performance.

Semiconductor layer

The uniformity of a semiconductor layer can be monitored using techniques like Atomic Force Microscopy (AFM) or other scanning probe techniques. Although these methods are very accurate, these methods are by design only applicable for very small sample surface areas. Therefore, for roll-to-roll manufacturing other, less accurate, large area methods are preferable for inline monitoring the quality of the semiconductor layers. A technique more suitable for adaptation for large area inspection of surface topology is optical/electron microscopy, *i.e.* Confocal Raman microscopy, interferometry, small angle X-ray scattering (SAXS), X-ray diffraction (XRD), TEM, *etc.* The chemical structure and electrical performance of the semiconductor can be assessed using techniques such as Fourier-Transform Infrared (FTIR) spectroscopy, secondary ion mass spectrometry (SIMS), Spreading Resistance Profiling (SRP).

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3 Main parameters for measurement and automation

Based on the research results on organic solar cells (VTT), organic thin film transistors (TNO/Holst), and electrochromic displays (Acreo), the following aspects of fabrication were found to be the current primary limiting parameters and are considered in more detail in the next section:

- Overlay and registration
- Layer uniformity
- Pattern quality (*e.g.* feature size, shape)
- Product functionality

3.1 Overlay and registration

3.1.1 OSC manufacturing and measurement requirements

Polymer solar cells typically have a multilayer structure, containing an anode, a hole injection/transport layer, a photoactive layer and a cathode. The output power of an organic solar cell is dependent on the active area and also related to the spectrum and intensity of incoming light. Single solar cell with certain size might produce enough energy to empower certain application, but the output voltage is often typically limited to 0.6 V. Therefore, these cells are required to be interconnected serially in order to increase the output voltage and form organic solar cell modules instead of single cells.

Front side barrier layer will most likely cover the whole substrate, so no registration is needed for front side barrier. The anode layer processed on top of front side barrier is either indium tin oxide or other corresponding material or metal grid and conductive organic polymer combination. The following layers including anode, hole transport layer, photoactive layer, interlayer, electron transport layer, cathode, contact pads and adhesive need to be aligned with each other. The registration requirements are highly dependent on the organic solar cell module design. It could be estimated that registration accuracy of 100-200 μm would be currently attainable. However, in a long term, the target would be less than 50 μm . The registration requirements for each layer in OSC structure are shown in Table 10. The registration requirements are dependent on the feature size requirements defined in the layout of the organic solar cell module.

Table 10 Registration requirements defined for layers in organic solar cell structure.

Layer	Value	Target	Unit
Anode (ITO)	100-200*	<50	μm
Metal grid	100-200*	<50	μm
Conductive organic polymer	100-200*	<50	μm
Hole transport layer	100-200*	<50	μm
Photoactive layer	100-200*	<50	μm
(Interlayer)	100-200*	<50	μm
Electron transport layer	100-200*	<50	μm
Cathode	100-200*	<50	μm
Contact pads/busbars	100-200*	<50	μm
Adhesive	100-200*	<100	μm

*highly dependent on the organic solar cell module layout

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The above-mentioned requirements for the registration should be measured with sufficient accuracy and precision. We believe that registration and overlay measurements should be *valid* within a range of $\pm 5\%$, which means that the registration and overlay should be measured with a combined accuracy and precision of $\pm 5\%$, resulting in the measurement requirements shown in Table 11.

Table 11 Measurement requirements defined for layers in the organic solar cell structure.

Layer	Value	Target	Unit
Anode (ITO)	5-10	<2.5	μm
Metal grid	5-10	<2.5	μm
Conductive organic polymer	5-10	<2.5	μm
Hole transport layer	5-10	<2.5	μm
Photoactive layer	5-10	<2.5	μm
(Interlayer)	5-10	<2.5	μm
Electron transport layer	5-10	<2.5	μm
Cathode	5-10	<2.5	μm
Contact pads/busbars	5-10	<2.5	μm
Adhesive	5-10	<5	μm

3.1.2 OTFT manufacturing and measurement requirements

In view of the overlay and registration measurements, two distinct features of the OTFT are important: The registration of the gate pattern with respect to the substrate and the overlay of the source/drain patterns with respect to the gate. If the source and drain pattern are applied separately, the mutual registration should also be considered. Table 12 shows the values taken from the requirements for manufacturing report.

Table 12 Overlay and registration manufacturing requirements for OTFT.

Feature	Dimension	Value			Unit
		Short term	Medium term	Long term	
Gate pattern	Registration	50	25	10	μm
Source/drain pattern	Overlay	5	2.5	0.1	μm

The abovementioned requirements should be measured with sufficient accuracy and precision. We believe that registration and overlay measurements should be *valid* within a range of $\pm 5\%$, which means that the registration and overlay should be measured with a combined accuracy and precision of $\pm 5\%$, resulting in the measurement requirements shown in Table 13.

Table 13 Required validity of overlay and registration measurements for OTFT.

Feature	Dimension	Value			Unit
		Short term	Medium term	Long term	
Gate pattern	Registration	2.5	1.3	0.5	μm
Source/drain pattern	Overlay	250	125	5	nm

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3.1.3 EC-Display manufacturing and measurement requirements

In view of the overlay and registration measurements, two distinct features of the EC display are important: The distance between the electrodes which should be as small as possible allowing high image resolution still giving a sufficient electric break (at least several MΩ) and the overlay/registration of the electrolyte. Also the thickness of the electrolyte is crucial to allow for a low total height of the device and enhance the possibility to good print resolution. Table 14 shows the values taken from the requirements for manufacturing report.

Table 14 Overlay and registration manufacturing requirements for EC-displays.

Feature	Value			Unit
	Short term	Medium term	Long term	
Distance between electrodes	100 - 200	50	25	μm
Electrode size	≥ 2 x 2*	≥ 1 x 1**	≤ 0.5 x 0.5**	mm
Electrolyte thickness	40	20	≤ 10	μm
Electrolyte positioning	± 100	± 25	± 5	μm

* Element based displays (pre defined image)

** Pixel based displays (matrix addressed, not pre defined image)

The abovementioned requirements should be measured with sufficient accuracy and precision. A video surveillance system capable of register fault sizes according to Table 15 below at production speed and over sufficient web width (or choosing critical spots) should be used. The system should have capacity to store the data for statistics and yield control.

Table 15 Required validity of overlay and registration measurements for EC displays.

Feature	Possible fault detection size for video surveillance system			Unit
	Short term	Medium term	Long term	
Distance between electrodes/electrolyte positioning	< 100	< 25	< 5	μm

3.2 Layer uniformity

3.2.1 OSC manufacturing and measurement requirements

The requirement for the layer uniformity in terms of layer thickness and surface roughness for organic solar cell component is shown in Table 16 and Table 17. Requirements for each layer are considered separately. These values are taken from D1.2 "Organic solar cell manufacturing requirements". In addition, requirements for the interlayer and electron transport layer are added to the table. These were not introduced in D1.2. The most challenging layers in a printing point of view are interlayer and electron transport layer since the layer thickness should be ~5 nm.

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Table 16 Layer thickness manufacturing requirements for OSC.

Layer	Value	Target	Unit
Anode (ITO)	>1	0.5-1	μm
Metal grid	200-500	100-200	nm
Conductive organic polymer	150-200	100-200	nm
Front side barrier	>10	1	μm
Hole transport layer	40-100	< 50	nm
Photoactive layer	80-300*	80-300*	nm
(Interlayer)	20	5	nm
Electron transport layer	20	5	nm
Cathode	1-10	~10	μm
Contact pads/busbars	1-10	~10	μm
Adhesive	2-20	> 1	μm

*dependent on the absorption maxima of the photoactive layer

Table 17 Surface roughness manufacturing requirements for OSC.

Layer	Value	Target	Unit
Anode (ITO)	> 100	< 5	nm
Metal grid	30-60	< 5	nm
Conductive organic polymer	<30	< 5	nm
Front side barrier		< 10	nm
Hole transport layer	1-30	< 5	nm
Photoactive layer	< 30	< 5	nm
(Interlayer)	< 10	< 1	nm
Electron transport layer	< 10	< 1	nm
Cathode	1	< 0.1	μm
Contact pads/busbars	1	< 0.1	μm
Adhesive	< 1	< 0.1	μm

The layer thickness requirements for different OSC layers were presented above. It is assumed that the $\pm 5\%$ accuracy and precision is needed to measure the layer thicknesses for each active layer. The required validity of layer thickness measurements for OSC is shown in Table 18.

Table 18 Required validity of layer thickness measurements for OSC.

Layer	Value	Target	Unit
Anode (ITO)	50	25	nm
Metal grid	10	5	nm
Conductive organic polymer	8	5	nm
Front side barrier	500	50	μm
Hole transport layer	2	2	nm
Photoactive layer	4	4	nm
(Interlayer)	1	0.25	nm
Electron transport layer	1	0.25	nm
Cathode	50	50	nm
Contact pads/busbars	50	50	nm
Adhesive	100	50	nm

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3.2.2 OTFT manufacturing and measurement requirements

In view of the layer uniformity, several features of the OTFT for display should be considered. Table 19 shows the values taken from the requirements for manufacturing report.

Table 19 Layer uniformity manufacturing requirements for OTFT.

Feature	Dimension	Value			Unit
		Short term	Medium term	Long term	
Gate pattern	Vertical	10	1	0.1	µm
Dielectric + modifier layer	Vertical	500	100	10	nm
Semiconductor layer	Vertical	1	0.1	0.1	µm
Source/drain pattern	Vertical	10	1	0.1	µm
Encapsulation layer	Vertical	250	100	50	µm

The abovementioned requirements should be measured with sufficient accuracy and precision. We believe that layer thickness measurements should be *valid* within a range of $\pm 5\%$, which means that the layer uniformity should be measured with a combined accuracy and precision of $\pm 5\%$, resulting in the measurement requirements shown in Table 20.

Table 20 Required validity of layer uniformity measurements for OTFT.

Feature	Dimension	Value			Unit
		Short term	Medium term	Long term	
Gate pattern	Vertical	500	50	5	nm
Dielectric + modifier layer	Vertical	25	5	0.5	nm
Semiconductor layer	Vertical	50	5	5	nm
Source/drain pattern	Vertical	500	50	5	nm
Encapsulation layer	Vertical	12.5	5.0	2.5	µm

3.2.3 EC-Display manufacturing and measurement requirements

The EC display is a robust and in its simplest form (element based lateral displays showing pre defined images) demanding rather relaxed printing parameters. Still when coming to pixel based displays and vertical built systems the requirements are sufficiently higher.

The element based lateral displays are basically consisting of two printed layers, PEDOT:PSS and an electrolyte. The thickness of the PEDOT:PSS is typically a few microns while the electrolyte is between 15 to 40 microns. The critical parameters are the electric break between the electrodes and the thickness of the electrolyte while pin holes etc in the printed areas are of minor influence.

When dealing with pixel based and vertically built systems the demands on the printing accuracy rises. To obtain a high fill factor the distances between the pixels must be kept very narrow still with a high electric resistance between them. The electrolyte must be

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well defined as thin as possible to reduce building height, pin hole free and not migrating outside its originally printed shape to avoid cross talk between the pixel elements

For the element based lateral displays the interesting parameters are the resistance between the display electrodes which should be as high as possible, preferably over 5 MΩ and the thickness of the electrolyte which should be as low as possible still giving a fast switch time. One method to measure the functionality of the display and thereby the influence of the above parameters is to measure the switch time the contrast and the retention time of the display. This can be done in an automatic measuring station of line, in line measurements is still to be developed.

For the pixel based and vertically built system high resolution video surveillance systems have to be to measure in line and inspect for registration possible shortcuts and pin holes. For controlling the thickness and uniformity of printed surfaces interference measurements (earlier described) can be used.

To control the properties such as adhesion, mechanical stability, migration of UV cured layers in for example a UV cured electrolyte you need to measure the UV dose. This can be done using a sensor within the UV lamp unit or using control stripes attached to the web to later be read out off line.

3.3 Pattern quality

3.3.1 OSC feature size manufacturing and measurement requirements

The feature size of the printed pattern is highly dependent on the used printing method. In Table 21, the feature sizes that could be attain with certain printing method is presented.

Table 21 Feature sizes of different printing methods

Printing method	Feature size [μm]
Gravure printing	(15) 20-75
Flexography printing	(20) 40-80
Offset printing	(10) 25-50
Screen printing	(50) 75-100
Inkjet printing	10-50

In Table 22, the feature sizes for each layer in OSC structure is shown. Besides the printing method, the particle size is critical parameter for printing electronic and optical materials. For instance, for printing of ITO nowadays feature size of 100 μm could be obtained. The small particle size of typically < 30 nm is a major requirement to reproducibly realize feature sizes on the sub-micron scale [Puetz, J., et al., Thin Solid Films, 516(2008)14, pp.4495-4501]. The requirement for the feature size is dependent on the organic solar cell module design. However, the feature size is not as critical as for instance in the case of OTFT. For other layers except metal grid, contact pads/busbars and adhesive, the target is on reaching a feature size below 50 μm. Feature size of <50 μm should be enough because larger areas are normally prepared in the organic solar cell structure instead of small features. High resolution (< 10 μm) and high conductivity is required for metal grid layer. The metal grid lines should be as thin

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as possible i.e. the grid should not inhibit the light to propagate and reach the photoactive layer in which the absorption of photons occurs. The feature size of busbars is defined $<25 \mu\text{m}$. Busbar should be a thin, high conductive line which forms a contact to anode and diminishes the impact of high resistivity of anode to the performance of large area organic solar cells/solar cell modules. Adhesive is either deposited as a continuous film or patterned on top of the OSC structure. Although the adhesive would be patterned, the feature size $<100\mu\text{m}$ should be reasonable.

Table 22 Feature size manufacturing requirements for OSC.

Layer	Printing/coating method	Value	Target	Unit
Front side barrier	Different coating methods	No patterning	No patterning	
Anode (ITO)	<ul style="list-style-type: none"> ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing ▪ Rotary screen printing 	> 100	< 50	μm
Metal grid	<ul style="list-style-type: none"> ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing ▪ Offset printing 	> 40	<10	μm
Conductive organic polymer	<ul style="list-style-type: none"> ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing 	> 100	< 50	μm
Hole transport layer	<ul style="list-style-type: none"> ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing 	> 100	< 50	μm
Photoactive layer	<ul style="list-style-type: none"> ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing 	> 100	< 50	μm
(Interlayer)	<ul style="list-style-type: none"> ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing 	> 100	< 50	μm
Electron transport layer	<ul style="list-style-type: none"> ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing 	> 100	< 50	μm
Cathode	<ul style="list-style-type: none"> ▪ Rotary screen printing ▪ Gravure printing ▪ Flexography printing ▪ Inkjet printing 	> 100	<50	μm
Contact pads/busbars	<ul style="list-style-type: none"> ▪ Rotary screen printing ▪ Inkjet printing ▪ Gravure printing ▪ Flexography printing 	> 100	<25	μm
Adhesive	<ul style="list-style-type: none"> ▪ Rotary screen printing ▪ Gravure printing ▪ Flexography printing ▪ Coating methods 	> 100	<100	μm

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The feature size requirements for different OSC layers were presented in the table above. It is assumed that the $\pm 5\%$ accuracy and precision is needed to measure the layer thicknesses for each active layer. The required validity of feature size measurements for OSC is shown in Table 23.

Table 23 Required validity of feature size measurements for OSC.

Layer	Value	Target	Unit
Front side barrier	No patterning	No patterning	
Anode (ITO)	> 5	< 2.5	μm
Metal grid	> 2	< 0.5	μm
Conductive organic polymer	> 5	< 2.5	μm
Hole transport layer	> 5	< 2.5	μm
Photoactive layer	> 5	< 2.5	μm
(Interlayer)	> 5	< 2.5	μm
Electron transport layer	> 5	< 2.5	μm
Cathode	> 5	< 2.5	μm
Contact pads/busbars	> 5	< 1.25	μm
Adhesive	> 5	< 5	μm

3.3.2 OTFT feature size manufacturing and measurement requirements

In view of the minimum feature size, several parameters of the OTFT for display should be considered. The most important parameters are registration and overlay. Table 24 shows the values taken from the requirements for manufacturing report.

Table 24 Relevant parameters for feature size from manufacturing requirements for OTFT.

Feature	Dimension	Value			Unit
		Short term	Medium term	Long term	
Gate pattern	Registration	50	25	10	μm
Source/drain pattern	Overlay	5	2.5	0.1	μm

The abovementioned requirements should be measured with sufficient accuracy and precision. More importantly, the required feature size (or critical dimension) of the is dependent on the most strict requirements for pattern positioning, *i.e.* the requirements for overlay of the source/drain patterns. We believe that the measurements should be *valid* within a range of $\pm 10\%$, which means that the layer uniformity should be measured with a combined accuracy and precision of $\pm 10\%$, resulting in the measurement requirements shown in Table 25.

Table 25 Required validity of feature size measurements for OTFT.

Feature	Dimension	Value			Unit
		Short term	Medium term	Long term	
Source/drain pattern		500	250	10	nm

3.3.3 EC-Display feature size manufacturing and measurement requirements

The requirements regarding pattern quality for the element based EC display as described above is rather relaxed. The display consists of two printed layers, the electrodes, PEDOT:PSS and an electrolyte. It is necessary to obtaining a good break

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without shortcuts between the electrodes meaning typically a distance between 100 to 200 μm . It is also important to position the electrolyte accurately over the electrodes a tolerance between 100 to 200 microns are acceptable depending of the application. Since screen printing often are used to produce these displays jagged edges have to be tolerated especially at angles around 45° in relation to the screen mesh. One issue is the possible migration of electrolyte outside the printed area which occurs over a period of time. For simple element based displays a certain migration (less than one millimetre) can be tolerated.

For the pixel based and vertical built displays the requirements are sufficiently higher. The distance between the pixels must be kept small, under 100 μm in the first generation displays. The edges must be straight and not jagged and the layer thicknesses must be low and pin hole free. Also the electrolyte must not migrate out from the printed area thereby avoiding short cuts and cross talk. This probably excludes screen printing as production method and also calls for material development.

Indirect measurements such as switch time, contrast and retention time can be used to indicate pattern quality as described above. For more specific measurements high quality video surveillance can be used for the element based displays these measurements should be sufficient.

For the pixel based and vertical displays video surveillance in combination with interference measurement should be used to control both lateral resolution and print quality as well as thickness (Z dimension) and pin holes.

3.4 Product functionality

3.4.1 OSC requirements and measurements

The basic organic solar cell electrical measurement composes of current-voltage characteristics in the dark and under illumination conditions. From the current-voltage characteristics several electrical key parameters are defined, such as open-circuit voltage (V_{oc}), short-circuit current (I_{sc}), fill factor (FF), maximum power point (P_m) and power conversion efficiency (PCE). The point in which the illuminated curve intersects with the x-axis is called V_{oc} . Correspondingly, the point in which the illuminated curve intersects the y-axis is depicted as I_{sc} . In the fourth quadrant, the organic solar cell generates power. P_m is the point in which the product of current and voltage is the largest. The FF is calculated by dividing the maximum power by the product between V_{oc} and I_{sc} . The power conversion efficiency (η) is calculated as follows:

$$\eta = \frac{P_m}{E \cdot A},$$

Where E [W/m^2] is the light irradiance under standard test conditions, and A [m^2] the surface area of the solar cell. Hence, the FF can also be calculated with the following expression:

$$FF = \frac{\eta \cdot E \cdot A}{V_{oc} I_{sc}}.$$

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The current-voltage curves of a solar cell are depicted in Figure 5. Dark is marked with black, and illuminated with red.

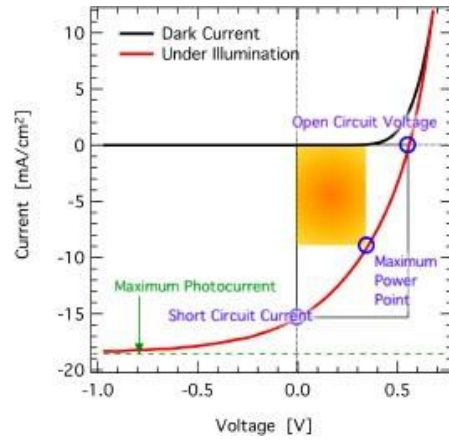
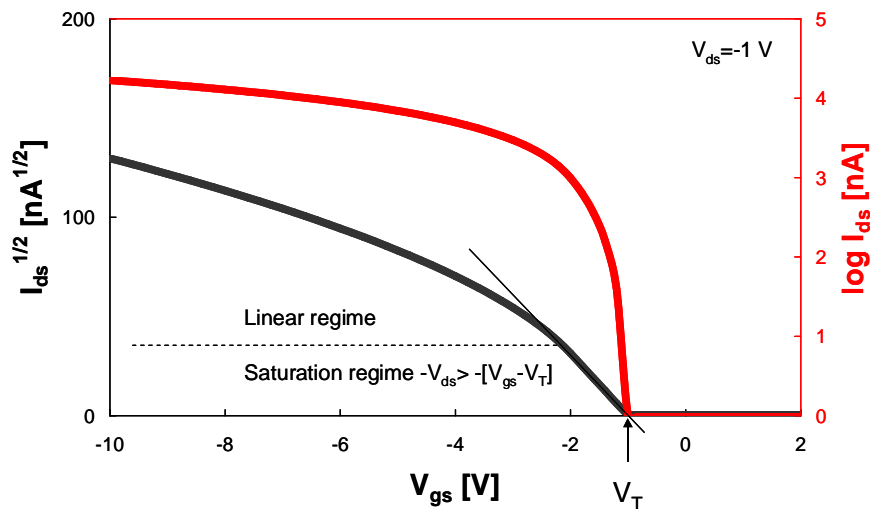


Figure 5 Current-voltage (I-V) curves of a solar cell (dark: black, illuminated:red) [www.blog.disorderedmatter.eu/]

Currently however, current-voltage measurements under illumination and under dark in a roll-to-roll process are not feasible. The electrical testing of organic solar cells will be performed in the assembly phase. The testing could be realised as follows. The current-voltage characteristics of the solar cell/solar cell module will be measured. The points that should be taken into consideration are V_{oc} , I_{sc} , P_m . The values in these points should be within defined limits. In a roll-to-roll process (inline measurements), instead of the product functionality, the focus is on inspection of the layer quality, pattern quality and registration.

3.4.2 OTFT requirements and measurements

The current-voltage (I-V) diagram shown in Figure 6, is an important tool to determine OTFT device performance.



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Figure 6 Example OTFT I-V response diagram.

In this diagram, the current between the drain and source (I_{ds}) is shown as a function of the bias voltage between the gate and the source (V_{gs}). This performance curve is valid for a certain fixed bias voltage between the drain and the source (V_{ds}). Several features of an OTFT can be derived from this diagram. See the report on OTFT for display manufacturing requirements for more details:

- On current (I_{on}); This is the current between the drain and the source when the OTFT is in the on-state.
- I_{on}/I_{off} ratio; This is the ratio between the drain-source current in the on-state and the leakage current between the drain and the source in the off-state.
- Threshold voltage (V_T); This is the voltage that discerns the conduction-state from the insulator-state. It can be determined in the linear or saturation operation regime (determined by V_{ds}) by linear regression of the I-V response diagram in the transition regime between conduction and insulation. The threshold voltage
- Hysteresis; Hysteresis can be determined by determining the I-V response diagram by varying V_{gs} from the on- to the off-state and *vice versa*. The hysteresis can then be determined from the maximum relative difference between the two curves.
- Sub threshold swing; This is the slope of the base-10 logarithm of I_{ds} versus the V_{gs} below the threshold voltage (defined by the linear intersection of a linear regression through the curve of the square root of I_{ds} versus V_{gs}).
- Carrier mobility. The slope of the I-V response curve near the threshold voltage can be used to determine the carrier mobility. For this purpose, the channel width as well as the channel length and area of overlap between the source/drain/gate should be available. Furthermore, the dielectric capacitance should be measured (*e.g.* by using a method described in Section 2.5.3). Although carrier mobility is considered an important parameter for OTFT device performance, we think that the before mentioned parameters are in fact the parameters that are most relevant to the end user of the OTFT.

Another parameter that can be determined from the I-V response diagrams is the switching frequency. This is done by determining the speed at which the OTFT switches state after changing the gate voltage from the on- to the off-states and vice versa. The requirements for the abovementioned parameters can be found in the report on OTFT for display manufacturing requirements and are repeated in Table 26.

Table 26 Electrical requirements for OTFT for display technology.

Electrical parameter	Value			Unit
	Short term	Medium term	Long term	
I_{on}	10^{-5}	10^{-6}	10^{-7}	A
I_{on}/I_{off}	10^4	10^6	10^8	
Gate voltage	100	50	10	V
Threshold voltage	5	1	0.1	V
Threshold voltage stability	10	5	1	%
Hysteresis	10	5	1	%
Sub threshold swing	N/A	N/A	N/A	V/Dec
Switching frequency	1	10	100	kHz

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We believe that electrical measurements should be *valid* within a range of $\pm 5\%$, which means that the properties in the table above should be measured with a combined accuracy and precision of $\pm 5\%$. Although the validity of the measurements in itself is not viewed upon as a difficult requirement to meet, these measurements will be very challenging in a large area roll-to-roll manufacturing environment where any measurement method is by preference contactless. It is not foreseeable that such methods will become available anytime soon and therefore we believe that real-time process monitoring methods for pattern and layer quality, combined with process control strategies such as SPC should be implemented to ensure proper end-of-line electric OTFT device performance (see Section 2.5.1 and 2.5.3).

3.4.3 EC-Display requirements and measurements

The overall requirements for an EC display are contrast, switch time and resolution. In the table below the expected parameters are listed.

Feature	Short term	Medium term	Long term	Unit
Contrast ΔL^*	15	20	25	
Switch time for 1 cm^2	< 10	< 5	< 2	Second
Resolution	Element based	25	50	DPI

The contrast measurements are done with a spectra photo meter using $L^*a^*b^*$ measuring mode only the L^* parameter is used. Switch time is determined by measuring charge transport in the display using a DAQ card and a lab view application. Resolution is determined when designing the display considering the design rules from time to time.

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4 Metrology and automation solutions

One of the major challenges for fabrication of polymer and printed electronics is to control the properties of functional layers, which have to be kept much more precise than with conventional colour printing. For example the thickness of gate dielectric layers strongly influence the electrical properties of organic TFTs and the resulting variation in electrical characteristics of single TFTs determines whether a polymer integrated circuit will work or not. In general high accuracy and uniformity is needed for functional layers.

Efficient process control is therefore a very helpful instrument to ensure these requirements and to increase yield in roll-to-roll manufacturing. Of course process control in a roll-to-roll manufacturing environment is much more complicated than in single substrate processing by two main reasons:

- The first difficulty is to find process control methods that are able to keep up with the high throughput targeted for printed and polymer electronics. This makes a new combination of precision and data acquisition speed necessary
- Secondly, process control has to be integrated inline with the production line, because it will be rather difficult and very inefficiently to take samples out of a web and measure them offline. Only very fast measurement methods are therefore suitable for this purpose, but offer also the possibility to control and adjust film parameters in a closed loop

A number of further requirements can be deduced for inline instrumentation:

- Equipment has to be robust to deliver consistent measurement values over a long time
- Acquisition times for inline measurements are limited or come along with an inaccurate positioning. It must be ensured that the demands on measurement accuracy can still be fulfilled
- In difference to offline measurements, where faults can be recognised and corrected quite easily, inline measurements must run mainly automatically. Therefore evaluation algorithms must be able to detect whether a measurement has been acquired under valid conditions
- Inline measurements are in continuous operation. Maintainability and exchangeability are therefore of high importance. The system should be therefore self-calibrating or furnished with an easy calibration possibility

4.1 Geometric inspection

4.1.1 Mechanical measurements

Requirements for mechanical measurements

Web tension is controlled using equipment like load cells, tension sensing rollers, tension breaks and clutches. To control the web tension is essential to keep the web from being elongated by stressing the material still having enough tension to get a good transport through the machine. This will mainly affect the web and registration in MD.

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Web guiding is used to control the web in TD. Usually it is used at the in feed of the material in the R2R printing machine before the first printing step and before the web tension control. You also want to use a web guide after a long drying oven to ensure correct positioning of the web in TD after a long loop out of control.

Thickness measurements of printed layers can be performed using equipment like Dektak or similar. Mechanical thickness measurements can be performed using mechanical thickness gauges (*e.g.* Checkline Europe, Dektak). The typical performance of mechanical thickness measurements are shown in Table 27.

Table 27 Typical performance parameters of mechanical thickness measurements

Parameter	Value			Unit
	Short term	Medium term	Long term	
Minimum thickness	10	1	0.1	µm
Accuracy	10	5	1	%
Precision	10	5	1	%
Area coverage	1	5	10	spots/s

Applications for mechanical thickness measurements

- MD registration
- TD registration
- Layer thickness, Z-levelling
- Measuring of the surface topography of screens “Rz” value to ensure good edge sharpness of printed lines.

4.1.2 *Acoustic measurements*

Requirements for acoustic measurements

Acoustic thickness measurements can be performed using time of flight gauges (*e.g.* DFT Instruments), the typical performance of acoustic thickness measurements are shown in Table 28.

Table 28 Typical performance parameters of acoustic thickness measurements

Parameter	Value			Unit
	Short term	Medium term	Long term	
Minimum thickness	10	1	0.1	µm
Accuracy	10	5	1	%
Precision	10	5	1	%
Area coverage	1	5	10	spots/s

The advantage of using an acoustic method that it can be contact free and thereby used on a moving web.

Applications for acoustic measurements

- Measurements of thicknesses of printed layers in- or off-line.
- Measurements of print forms such as screens. Establishing the EOM (emulsion over mesh) value which is important to ensure good uniformity of printed surfaces.

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4.1.3 SEM/AFM measurements

Requirements for SEM/AFM measurements

A SEM, Scanning Electron Microscope, is scanning the sample surface with help of electron beam. The electron beam gives information about the samples topography and other properties like which materials the sample consists of. To determine material content the SEM should be equipped with EDX to be able to look at the Röntgen spectra and find the peaks for different materials. The samples need to be conducting, not volatile and heat resistant; they also need to be vacuum compatible. If the samples are not conducting they must be covered with a metal, usually gold, by sputtering or evaporation. The detectors in the SEM can be of different kind e.g. “in lens”, “secondary ion” or “back scatter”. Different detectors give images with different characteristics e.g. the back scatter (BSE) detector detect areas with different chemical compositions.

The finest resolution in X/Y direction is in practical use down to ten nanometers, the SEM give no value on the Z topography but indicates variation in height. By tilting the sample three dimensional images and topography can be established. Enlargement can be chosen from 50 to 500000 (1000000) times and the inspected area in relation to enlargement cm^2 to μm^2 . The method is fast with no extensive sample preparation and the equipment medium priced.

AFM Atomic Force Microscope, uses a cantilever with a tip that explores the topography of the sample, it usually works in tapping mode, the cantilever is oscillated and tapping the surface a laser beam is reflected on the back side of the cantilever and the reflected beam and thereby the height of the cantilever is detected by an array of photodiodes. The probe tip of the cantilever does actually not touch the surface of the sample rather is the near surface forces like the van der Waals forces measured when the tip is close to the surface. The AFM can register topography (X/Y/Z), and is tapping the surface 512 to 1024 times per measuring line. The measured area is from 100 by 100 μm down to 500 nm. The resolution in Z direction is down to atom size. AFM can utilize all samples that are not liquid or sticky the samples do not need to be conducting and there is no vacuum used. The method registers topography, different materials and solidity of the sample. The measurements are more time consuming than when using the SEM but the sample preparation is kept to a minimum.

Applications for SEM/AFM measurements

For SEM conducting non volatile samples should not be sensitive to vacuum conditions. If the samples are non-conducting deposition of a conduction layer onto the sample is necessary. The coverage with metal will hide fine details in the sample, 3D images possible by tilting the sample. For AFM not liquid or sticky surfaces, no vacuum, minimal sample preparation, 3D imaging is standard.

Items with thin printed layers like OTFT:s and Organic solar cells are suitable for inspection by SEM and AFM. EC displays though, have a thick layer of watery electrolyte and are therefore not suitable for these methods.

4.1.4 Optical measurements

Requirements for optical measurements

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To implement optical measurements in an inline manufacturing environment a number of requirements have to be fulfilled. The constructive integration normally causes not too much problems. Ideally, the measurement head is mounted above the web, preferably near a tensioning roller. For measuring transmission an additional optical detector is mounted symmetrically underneath the web. Requirements on space, stability and mounting precision are not hard to meet.

Spatial resolution

Diameters of the measurement spot in the range of 1 to 10 mm can be put into practice with reflection, transmission or ellipsometric measurement systems. In ellipsometers an elliptic shape of the measurement spot (with a ratio 2-3 of the semiaxes) has to be taken into account. With adequate optics the measurement spot can be reduced to 0.1 mm. Below this size a loss in the usable wavelength spectra has to be accepted, especially in the non-visible area, because an achromatic optical systems can be hardly achieved anymore.

Most robust measurement system is the reflectometry with its nearly vertical angle of incidence, which makes the system tolerant against vertical distance variations and keeps the lateral measurement position constant

Web speed and resolution in web direction

To get a spatially resolved measurement from a moving web, the web should not move more than 10% of the measurement spot diameter during the typical measurement time. With a spot diameter of 10 mm and a typical measurement time of 100 ms a maximum speed of 0,6 m/min can be tolerated. Therefore an optical measurement with continuous illumination is limited to rather low web speeds. To extend the process control method to higher web speeds the use of a flash illumination, e.g. with Xenon flash lamps, may be used. With this method measurement times down to 10 μ s seem to be achievable and correspondingly web speeds up to 600 m/min can be assessed.

Resolution in transverse direction – multi channel systems

In many cases it may be sufficient to control a coating at one fixed position in the transverse direction and to monitor the coating properties in web direction. However, for some applications it will be necessary to control also the uniformity in transverse direction. This can be achieved in different manner. Most simple approach is to integrate multiple measurement systems on several places perpendicular to the web direction. Of course, this also multiplies the costs of the process control system.

Another approach is a measurement head that can be moved perpendicular to the web and therefore can be used to scan the transverse direction. The advantage is that the process control system requires only one measurement head. In continuous web processing this approach leads to a superposition of the moving measurement head with the web movement and therefore may limit the resolution. It therefore will be best suited for stop and go roll-to-roll systems.

When neither parallel nor scanning systems are applicable, a multiplex configuration may be a solution, which uses the most expensive components commonly. This can be achieved by an optical multiplexer (see Figure 7) that deflects the optical beam to the measurement position. The switching times are normally faster than positioning in a corresponding scanning system.

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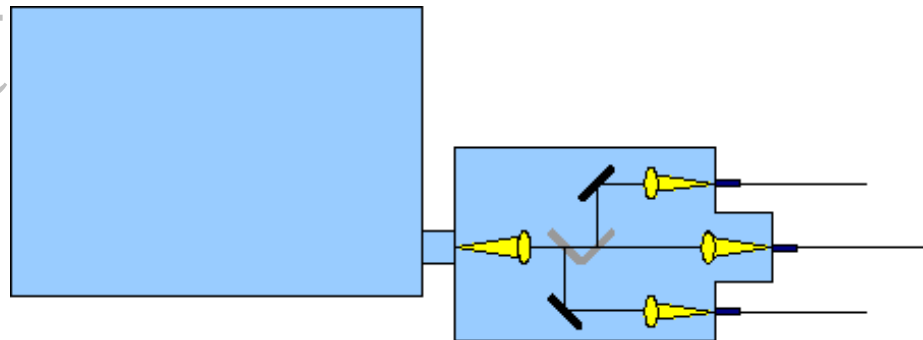


Figure 7 Optical multiplexer for assessment of different measurement positions with one spectrometer.

Applications for optical measurements

Thickness measurements

Typically thickness is determined by measuring the step height of a patterned coating by scanning the surface profile of the sample either by a stylus or focussing a laser beam on the surface. However in view of continuous manufacturing, especially reel-to-reel processing, the application of this method is quite restricted because it is rather slow and not usable for inline fabrication.

An alternative to scanning thickness measurements are optical measurements that can be used on unstructured coatings provided that these are sufficiently optical transparent. Generally optical measurements are based on interferences that change an initial spectra in a reflected or transmitted light beam when a substrate is coated with a thin layer. Mostly the method is used for thickness measurements, but it can also be used to characterize other surface properties like roughness, composition or crystallinity.

In optical thickness measurements, a wave-length dependant analysis of a reflected or transmitted optical beam, which is influenced by the optical behaviour like adsorption in the material and interferences originating from the material interfaces, is used. As the optical parameters are very specific to a material system the resulting spectra can be fitted to calculated data and among other parameters the thickness of a coating can be extracted. The method works in a rather broad thickness range from nm to several microns as far as the materials show sufficient transparency. In principle it can not be applied only to single coatings, but also to determine multilayer stacks.

To apply this method a calibration of the material stack has to be done in advance, which means that optical parameters, especially index of refraction and adsorption coefficients, have to be measured for each material that is implicated. Normally this is done on layers with known thickness by spectral ellipsometry.

Optical measurements are an indirect type of measurement using more or less complex mathematical operations to calculate the result. Spectral changes are not easy to interpret, therefore a successful measurement implementation in an inline processing environment depends essentially on the robustness, simplicity and predictability of the used algorithms in the numerical fitting procedure

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The figure below shows as example for the method by measuring a stack of organic semiconductor and gate dielectric on a PET foil. The transmitted spectrum shows the adsorption of the PET substrate below 320 nm, the adsorption edge of the semiconductor at 440 nm and the interferences originating from the gate dielectric. The best-fit calculation that matches the spectrum gives rather precise values for this two layer system.

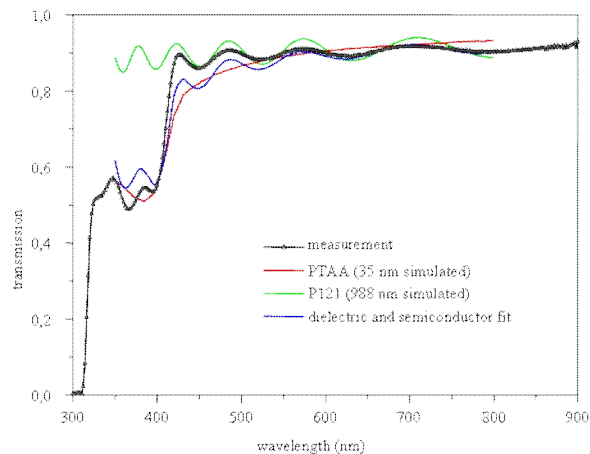


Figure 8 Transmission through a web coated with organic semiconductor (PTAA) and dielectric layer and calculated constituents.

The typical performance of optical thickness measurements are shown in Table 29:

Table 29 Typical performance parameters of optical thickness measurements

Parameter	Value			Unit
	Short term	Medium term	Long term	
Minimum thickness	10	1	0.1	µm
Accuracy	10	5	1	%
Precision	10	5	1	%
Area coverage	1	5	10	spots/s

4.2 Mechanical inspection

The parameters that traditionally are controlled in the printing process are:

- In feed registration (if the substrate is patterned before roll-to-roll processing)
- Registration between layers
- Steering of the web in cross web direction (web guiding)
- Web tension

For functional printing there is also a need to measure for example resistance in conducting lines and even to do functionality tests of devices in line. This can of course be achieved by taking samples from the roll after printing but this is time consuming and requires re winding of the roll. If samples are taken from the roll after the printing it

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is not possible to do in line printing adjustments. If a fault is detected after the printing is finished, there is a risk that a lot of expensive material is wasted. It is therefore preferred to do the functionality tests in line.

Feature	Short term	Medium term	Long term
In feed registration	Yes	Yes	Yes
Registration between layers	Yes	Yes	Yes
Web guide	Yes	Yes	Yes
Web tension	Yes	Yes	Yes
Resistance measurements	No	Yes	Yes
Functionality controls	No	No	Yes

4.3 Chemical-physical inspection

4.3.1 Spectroscopy (pollution, doping)

Near infrared spectroscopic measurement

Even spectroscopic absorption measurement can be for example used to measure film thickness, the near infrared spectroscopic measurement is not so usable in contamination measurement. This is due to the accuracy of the online spectroscopic measurement, which is typically in the area 0.1 – 3% (solid content or by volume), where 0.1 % accuracy is very difficult (rarely) to achieve.

4.3.2 Ambient O₂ and H₂O measurements

The measurement of ambient oxygen and moisture content is highly important when manufacturing sensitive electronic components by printing. Some materials are sensitive to oxygen and moisture, thus decreasing the component lifetime. In addition, the manufacturing stages are sensitive to the moisture and oxygen level changes during the process, thus making the control and adjustment of the oxygen and moisture level extremely important.

Oxygen sensors

Oxygen sensors are able to measure the proportion of oxygen gas (O₂) in the ambient air/gas. The measurements can be done continuously on-site, thus making them suitable for the printing electronics processes. Oxygen sensors have typically semi-permeable films that pass through oxygen. This diffusion initiates chemical reactions and changes the properties of the sensing material as a function of oxygen level. The most commonly used sensor types are electrochemical, paramagnetic, polarographic, and zirconium oxide analyzers. Other analyzers use infrared, ultrasonic, or laser to detect oxygen.

Electrochemical sensors (see Figure 9) have two dissimilar metal electrodes immersed into an aqueous electrolyte. Oxygen diffuses through a diffusion barrier onto the cathode where they are reduced to ions. These ions migrate through the electrolyte to the anode and become oxidized. The reduction and oxidation reactions generate an electric current proportional to the oxygen level. Electrochemical sensors have a simple structure, wide detection range, and low cost. However, the lifetime is limited and cross-sensitivity to other gases interferes with the measurement. The operation principle

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The principle of polarographic oxygen sensors is similar to that of the electrochemical sensors except that oxygen ions flow from the anode to the cathode.

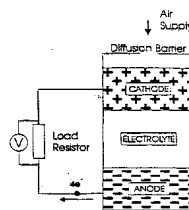


Figure 9 Structure of an electrochemical oxygen sensor. [http://www.habmigern2003.info].

Paramagnetic sensors contain a pair of nitrogen-filled glass spheres which are suspended by a thin wire within a magnetic field. As oxygen molecules having high magnetic susceptibility pass through the sensor, they are attracted towards magnetic fields. As a result, the magnetic field is strengthened and the spheres suspended from the wire starts to rotate. The degree of rotation is measured and the electric current required to maintain the spheres in their normal states is proportional to the oxygen concentration. Paramagnetic sensors are accurate in the range of 1-100 % and have fast response time and long lifetime. The disadvantages are expensiveness and sensitivity to other magnetic gases.

Zirconium oxide sensors use a solid zirconium oxide electrolyte which is contacted by gases on both sides: on one side by a reference gas and on the other by the measured gas. The sensor is heated to 650 °C to form a porous zirconium lattice that allows oxygen flow from higher concentration to lower one. This produces a voltage between the electrodes whose magnitude is proportional to the partial pressure difference of the oxygen between the electrodes. Zirconium oxide sensors have a wide measurement range and fast response time but their lifetime is rather short due to the cross-sensitivity to other gases and the high temperature. The principle of the sensor is illustrated in Figure 10.

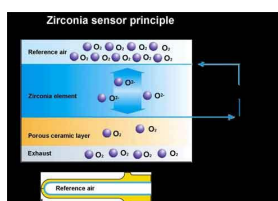


Figure 10 Principle of zirconium oxide oxygen sensor. [http://www.sparkplugs.com]

Infrared sensors rely on the changes of the absorption of infrared wavelengths with gas concentration whereas ultrasonic sensors utilize ultrasonic transducers that are able to produce and detect ultrasonic signals that propagate through the sensing gas, bounce-off from oxygen molecules, and return to the detector. Laser sensors use absorption spectroscopy to measure oxygen levels. The changes in the oxygen concentration attenuate the laser beam intensity. Laser sensors are highly accurate and sensitive since no other gases absorb light at the same wavelengths as oxygen.

Humidity sensors

Hygrometers measure the relative humidity (RH) by measuring changes in the properties of the sensing layer caused by the moisture. The most common hygrometer

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types used in the industrial process control are capacitive, resistive, thermal conductivity, and dew-point sensors. These sensors analyze changes in electrical or thermal properties of the sensing layer but there are also sensors that sense changes in weight, volume, or transparency of the materials. The choice of the sensor technology depends on the environmental conditions encountered during the manufacturing process.

Capacitive moisture sensors consist of a substrate onto which a thin film of dielectric material is deposited between two conductive electrodes. The upper electrode is porous to allow water diffusion into the sensing layer and to protect the sensor from contaminants. Figure 11 shows the structure of the capacitive moisture sensor. The absorption of moisture into the sensing layer changes its dielectric coefficient and the capacitance of the sensor. These sensors have a wide measurement range, good chemical vapour resistance, and ability to function also at high temperatures and are fully recoverable. The main limitation is the small capacitance change with moisture absorption (0.2–0.5 pF for a 1% RH change).

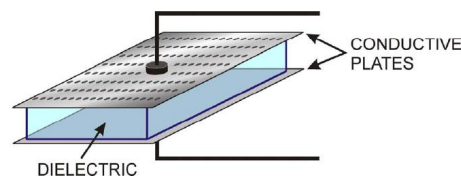


Figure 11 Structure of the capacitive moisture sensor. [Bull, K., ISHM 2006, p. 4]

Resistive moisture sensors, shown in Figure 12, have metal electrodes coated with a thin layer of hygroscopic material. As water is absorbed into the sensing layer, ions are formed that change the conductivity and impedance of the sensor as a function of moisture content. Resistive sensors are interchangeable, cost-effective, and stable. In addition, no calibration is required. However, the operation temperature is limited and contaminants decrease the lifetime.

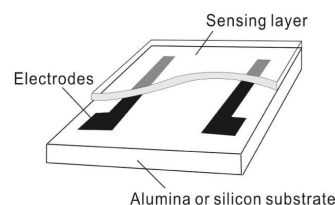


Figure 12 Resistive moisture sensor. [Packirisamy, M., et al., Sensor Review, 25(2005)4, pp. 271-276]

Thermal conductivity humidity sensors contain two thermistors one of which is encapsulated in dry nitrogen and the other is exposed to ambient conditions. Electric current is passed through the thermistors to increase their temperature after which the heat dissipation rates of the thermistors are measured. The rates differ because of the differences in the thermal conductivity of water vapour and nitrogen. The heat dissipation changes the thermistor resistance and the resistance difference is proportional to the RH. The sensor performs well in corrosive environments and at high temperatures but the sensor accuracy depends on the ambient temperature and the cross-sensitivity is high.

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Dew-point hygrometers contain typically a chilled polished metal mirror together with optoelectronic mechanisms. During chilling, the moisture condensation onto the mirror surface is detected and the temperature at which the condensation begins is the dew-point. These sensors are expensive but highly accurate RH measuring devices.

4.3.3 Surface energy

Contact angle measurements

Contact angle measurement is typical method to measure surface energy. Usually this measurement is done at laboratory, but VTT has studied that in principle the measurement can also be done online. With online contact angle measurement essential problems are

- Settling of the drop in the moving web
- Image acquisition timing (depth of the field is very low for microscopy imaging)
- Electric charge of the moving web (affects place where the drop is falling)

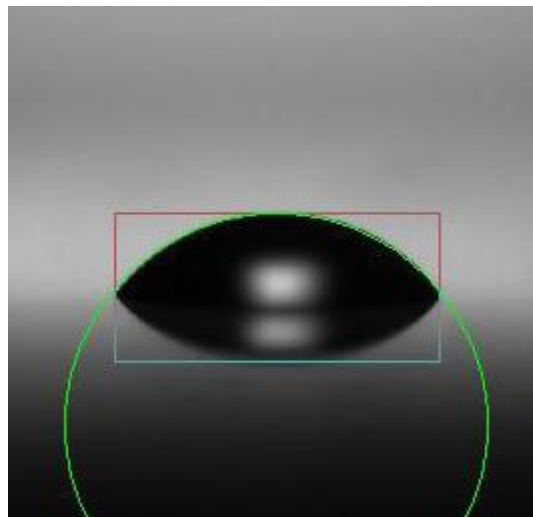


Figure 13 Water drop and contact angle calculation by machine vision.

In practice, online measurement is done as follows: Before the measurement, a point dispenser deposits fluid drops at the side of the web. The drop then moves along with the web towards the camera measurement point. There drop is detected with a laser beam, which then triggers an adjustable delay, which in turn triggers the camera. From the retrieved picture, the contact angle is calculated using machine vision software.

4.3.4 Viscosity

Viscosity is a measure of the internal flow resistance of ink. The ink viscosity can be determined either off-line or on-line. On-line measurements are often done in ink tanks/reservoirs using viscometers to determine the viscosity changes during a print run and to control the process. During the printing process, ink encounters high shear conditions, for example in ink transfer points, which affect the ink viscosity but this viscosity cannot be measured on-line. Therefore, off-line measurements of the dynamic viscosity are used to simulate the conditions the ink encounters during the printing process. This viscosity is measured with rheometers able to simulate different shear rates and forces, temperatures, and times.

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Viscometers are low-cost, simple instruments offering portability for remote testing and on-line measurement capability. However, these devices cannot measure low viscosity values due to their limited speed and torque. The measurement range is typically $0.1-10^3 \text{ s}^{-1}$. Rheometers, for their part, are more expensive but they can simulate the dynamic processes, thus offering a complete material characterization (viscoelasticity, flow properties). The low friction air bearings allow the measurement of low viscosities, too. The measurement range is extended to $10^{-6} - 10^5 \text{ s}^{-1}$ corresponding to the shear rates in printing.

Viscometers

Viscometers measure the ink viscosity under one flow conditions. In general, either the ink flows through the measurement head or vice versa. The drag caused by the relative motion of the ink and the measurement head can be used to determine the static ink viscosity. Viscometers can be divided into capillary, orifice, rotational, falling ball, vibration, and ultrasonic viscometers.

Capillary viscometers are most widely used to measure the viscosity of Newtonian inks. The volumetric flow rate of the ink through a fine capillary is measured by noting the time required the ink to pass through two graduation marks. Either gravity or external force is used to make the ink flow through the capillary. With external force, the ink is forced through the capillary at a predetermined rate and the pressure drop across the capillary is measured. It is also possible to use on-line capillary viscometers where a metering pump that diverts a small proportion of the ink flow in bypass via a precision capillary slot (Figure 14). The volumetric flow through the capillary is used to measure the viscosity.

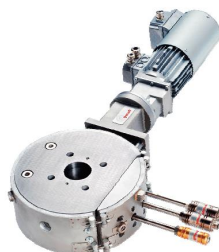


Figure 14. Gneuss in-line viscometer. [<http://www.gneuss.de>]

Orifice viscometers are also called efflux viscometers. They consist of a reservoir with an orifice. The time for the flow of a fixed ink volume through the orifice is measured, thus giving an arbitrary measure of the ink viscosity. There are several commercially available and standardized cups for this viscosity measurement.

Vibration viscometers are best suited for on-line viscosity measurements since they measure the damping of an oscillating resonator immersed directly into the ink. The higher the viscosity, the more power is required to maintain desired amplitude, the faster the decay of the oscillation or the larger the frequency change for a given phase is. Vibration viscometers are also suitable for measuring high-viscosity inks.

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Rotational viscometers measure the rotation rate of a solid shape in ink upon application of a known force required to initiate the rotation. The most common type is cone and plate viscometer where a cone of very shallow angle is in bare contact with a flat plate. In falling ball viscometers, a solid body is allowed to fall under gravity through the ink. After the initial acceleration, the body attains its terminal velocity that is measured and used to calculate the viscosity. Ultrasonic viscometers provide instantaneous and continuous measurement of viscosity by means of ultrasound that oscillates a sphere in ink.

Rheometers

Rheometers are able to measure the ink flow in response to applied forces, i.e., the dynamic viscosity and ink rheology. These devices can apply oscillatory and rapid step changes in stress and strain. The most common rheometer types are shear and extensional rheometers. Shear rheometers are further divided into capillary, rotational, and cone and plate rheometers.

In capillary rheometers, ink is forced through a capillary under laminar flow. Either the flow rate or the pressure drop across the capillary is fixed and the non-fixed parameter is measured. By varying the pressure (shear stress) or flow (shear rate), a flow curve can be determined and viscosity measured. The temperature of the viscometer is controlled. The shear rate can be varied from near 0 to 10^6 s^{-1} by changing the capillary diameter and applied pressure.

Rotational cylinder rheometers have two cylinders inside one another. The ink is placed within the annulus of the cylinders. One of the cylinders is rotated at a predetermined speed to determine the shear rate. The ink tends to drag the other cylinder and the force exerted on that cylinder is measured (shear stress).

Cone and plate rheometers (Figure 15) are the most common. The ink is placed on a horizontal plate and a shallow cone is placed into it. The ink is contained in the narrow gap between the cone and the plate. The small angles produce an approximately uniform shear rate through the sample when the cone is rotated. Typically, the cone is rotated and the torque transmitted to the plate through the ink is measured. However, these devices require calibration with known fluids. Cone and plate rheometers can also be operated in an oscillating mode to measure elastic properties, or in combined rotational and oscillating modes. In Weissenberg rheogoniometry the movement of a cone is resisted by a thin piece of metal which twists (torsion bar). The known response of the torsion bar and the degree of twist give the shear stress while the rotational speed and cone dimensions give the shear rate.

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Figure 15. Cone and plate rheometer. [<http://www.anton-paar.com>]

Extensional rheometers measure the extensional properties of the ink flow, such as elongation. This viscosity is commonly measured from materials subjected to tensile deformations. The development has been slow due to the challenges in the generation of homogeneous extensional ink flow. There are many commercially available extensional rheometers for different viscosity ranges. For low viscosity inks (< 1 Pa·s), capillary breakup, opposed jet, or contraction rheometers are used. With higher viscosity inks, filament stretching and constant-length rheometers are used. For example, in capillary breakup rheometers, ink is placed between plates which are rapidly stretched to a fixed level of strain. The ink filament necking and breakup are monitored as a function of time. There are also acoustic, falling plate, and capillary/contraction flow extensional rheometers. Acoustic rheometers measure the sound speed and attenuation of ultrasound that can be converted into ink compressibility and viscosity.

4.4 Functional inspection

The available tools for roll-to-roll functional inspection of the electronics products are very limited. Because of the roll-to-roll manufacturing approach and the fragile substrates involved only non-contact inline testing methods are considered as a viable option. This excludes mechanical solutions such as moving, shorting rubber, and direct (bed of nails) probe testing. Examples of non-contact tools found in the research were capacitive sensors for line short localization measurements (*e.g.* Nidec Read), electron beam array testing (Applied Materials, Shimadzu) for TFT for LCD display testing. In Section 2.5.3, some methods that enable sub-device electrical characterization are also discussed. Still, these methods require the application of control voltages to the devices under test via control pads and are therefore not completely non-contact. As concluded in Section 2.5.1 and 2.5.3, it is expected that inline inspection and control will eventually focus on process parameters rather than product parameters.

At the present level of maturity of the roll-to-roll manufacturing approach, functional inspection of the product parameters is vital for managing yield. To facilitate functional inline inspection, a stop-and-go manufacturing strategy may be implemented. In this approach, mechanical probe methods may be used as well. Because a stop-and-go manufacturing strategy may not be compatible with certain coating techniques an offline or end-of-line functional inspection step may be implemented instead. In offline testing, the manufacturing process may still be roll-to-roll based, but the rolls are transferred to a separate testing facility and then back to the manufacturing process.

For a new product, the design of the electronics circuit should incorporate facilities for off-line or end-of-line functional inspection, *i.e.* design for testability (DFT). This can

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be done in the form of additional monitoring pads that are used/sacrificed for testing purposes only, or by implementing test circuits that enable testing of clusters of components. Furthermore, the use of monitoring circuits, an analogue to the use of monitoring wafers in producing silicon based electronics, may be considered. In this approach, part of the products at different locations on the web is used/sacrificed for testing purposes only. By collecting statistics on the relations between product functionality and key process parameters, such as web tension, registration, overlay, layer uniformity, ambient temperature and pressure, and so on, a knowledge base can be generated to optimize end-of-line yield by optimizing process parameters. This way, the inline monitoring of product functionality can gradually be replaced by inline process monitoring.

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5 Conclusions

Based on the analysis of overlapping manufacturing requirements for the three applications considered in this study, *i.e.* organic solar cells (OSCs), electrochromic displays (ECDs) and organic thin-film transistors (OTFTs) for displays, the following main parameters for measurement and automation were identified:

- Overlay and registration
- Layer uniformity
- Pattern quality (*e.g.* feature size, shape)
- Product functionality

In view of the three applications, the measurement and automation solutions for overlay and registration should be able to resolve scales down to 0.1 μm precisely to meet ensure that the long term application requirements are met. On the medium term, measurement and automation solutions should be able to resolve scales down to 1 μm . For the short term applications overlay and registration should be valid within 5-10 μm . The requirements are most strict for the measurement and automation solutions for OTFT for display.

For layer uniformity, the short term requirements are already very strict for OSCs. The measurement and automation solutions should be able ensure layer thickness to be valid within 1-5 nm. For OTFT the short term requirements is in the order of 50 nm. For the long term applications, layer uniformity should be valid with in down to 0.5 nm.

Pattern quality is difficult to quantify and depends on the combination of the design of the application and the chosen manufacturing process. One important parameter considered here is critical dimension. The critical dimension requirements are most strict for the OTFT for display applications, resulting in short term requirements for measurement and automation in the order of 500 nm down to 10 nm for the long term applications. For OTFT this quantity is of course related to the requirements for overlay between the gate and the source/drain electrodes. For OSC, the critical dimensions are most important for the metal grid for capturing the charge carriers, resulting in requirements for measurement and automation solutions to resolve critical dimensions down to 0.5 μm for the long term applications.

At present testing product functionality, either online or offline, is vital for ensuring proper end-of-line yield for full organic electronics applications on flexible substrates. The used materials are expensive and are outperformed by silicon alternatives. Also, the roll-to-roll manufacturing technology for electronics requires improvements in terms of control and speed. Preferably, non-contact testing methods are used because these are compatible with roll-to-roll manufacturing techniques than mechanical probing. In case of mechanical probing, specific metering features could be added to the electronic design to avoid damage to the functional parts. As the manufacturing technology and a certain application mature, functional testing can and, in view of increasing production capacity, should be replaced by process based measurement and control solutions. Advanced process control solutions such as statistical process control (SPC), can then be used to optimize end-of-line yield and manufacturing speed.

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6 Literature

- ¹ Handbook of Silicon Semiconductor Metrology, A. C. Diebold, Editor, Marcel Dekker Inc., 2001
- ² Semiconductor Manufacturing Handbook, H. Geng, Editor, McGraw-Hill, 2004
- ³ Printed and thin film transistors and memory, P. Harrop and R. Das, IDTechEx, 2007.
- ⁴ International technology roadmap for semiconductors – Metrology, 2009.