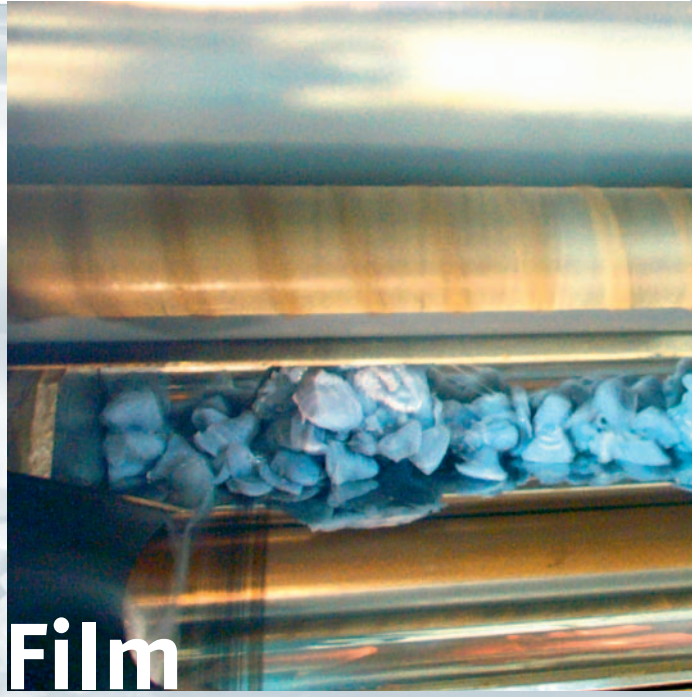


Comprehensive Concept. This article discusses defects that may possibly occur during production of film on calendaring equipment as well as the sources of black spots, their detection, evaluation and possible means of prevention.

High-Quality Pharmaceutical Film



ANDREAS SCHNABEL
OLIVER HISSMANN

Pharmaceutical products are packaged in plastic blister packs around the world. Every customer expects that the film used satisfies increasingly stringent requirements. In addition to economy, hygiene, protection in different climates and atmospheres, this also means a guarantee of maximum safety for the product packaged in compliance with new and ever more demanding requirements.

The monolayer film that is produced primarily on calendaring equipment is converted into blister packs or serves as the substrate for high-quality coatings and laminates.

These calendared monolayer films are plasticizer-free rigid PVC films that are manufactured in various thicknesses and colors. They can be classified into three product groups: With their good thermoforming characteristics and their good chemical resistance, homopolymer films are well-suited for production of standard blister packs. For demanding blister geometries or maximum processing speeds, copolymer and high copolymer films offer greatly improved flow characteristics.

Film Production by Means of Calendaring

To produce rigid PVC film, 4- or 5-roll L-calenders are usually employed. The calender performs the function of converting the highly viscous formulations into endless webs with a defined thickness and width. A calendaring line consists of a sequential arrangement of individual pieces of equipment – a material feed unit with gravimetric metering, a mixing section with heated and cooled mixing units, continuously operating co-kneaders or extruders, the calender itself with downstream equipment such as take-off and chill roll stand as well as a finishing section with longitudinal slitter and turret winder or cross cutter to produce rolls or cut-to-size products (Fig. 1).

With regard to processing, usually the high-temperature calendaring process is employed, since it generally offers a variety of possible settings for the polymer formulation and equipment through the use of highly effective stabilizers that permit formation of a rolling bank between the pairs of rolls and thus a higher temperature level.

The low-temperature process is used today almost exclusively for production of thin stretched film (for tape) with high values of mechanical properties.

Surface Defects During the Calendaring Process

One inspection attribute is based on defects that affect the visual appearance of

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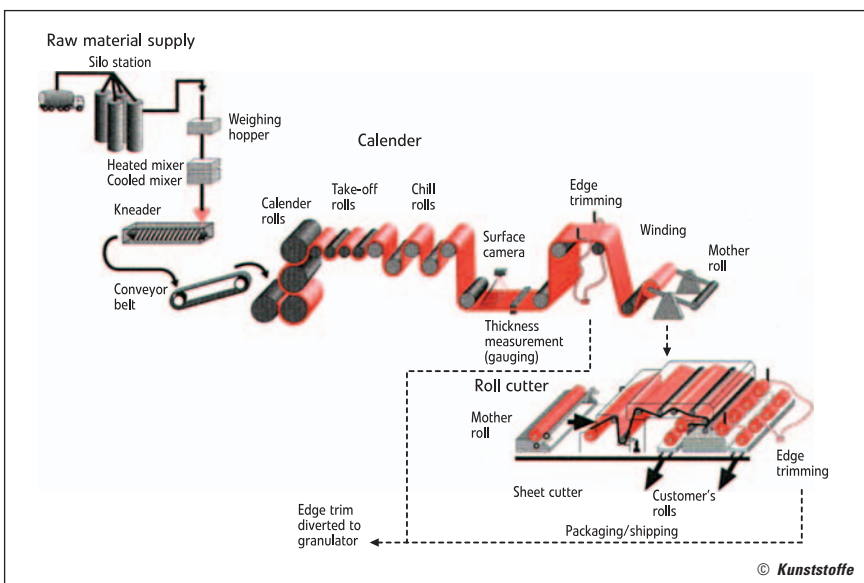


Fig. 1. Illustration showing production of rigid PVC film on a 4-roll L-calender



Material filling
the first nip of
a calender line

a product. Accordingly, a film defect is defined as a local deviation from the optical characteristics of a defect-free web. The physical effects indicative of a defect include different scattering behavior, less or more reflection and sometimes reduced or enhanced transparency.

The different terms often used to describe the defects can, for instance, be classified as follows – with reference to the transmission of light through transparent products (Fig. 2):

Specks / Material Specks: Are generally unprocessed material particles, air entrapped during processing (degassing, air introduced during the process), optical changes resulting from impressions made by the rolls, microstructures or deposits – not completely absorbing in terms of their optical behavior (light transmission).

Black spots / Fibers: Are generally material particles that have been damaged or burned as the result of thermal degradation, also foreign matter such as fibers, dust or other particles – largely opaque in terms of their optical behavior.

Holes: Are generally process-induced defects which occur in combination with specks and black spots that indicate tearing of the web or severe thinning – completely or almost completely transparent in terms of their optical behavior.

Flow lines, Burns, Structures: Are generally process-induced optical structures resulting from unequal material thickness (viscosity during processing, relaxation), thermal degradation (stabilization in PVC), or from deposits (structure-forming features on the surface of rolls or tooling), purely external defects or dripping fluids such as water/production residues – absorb only slightly in terms of their optical behavior –, but often can be detected only when viewed at a different angle and with special filters.

In the worst case, the film defects listed can lead to disruption of production by causing problems in subsequent processing steps. They are not only indicative of lower optical quality, but can also be the cause of equipment shutdowns or defective end products.

A reduction in quality that often occurs during the calendering process (in terms of the customer's requirements for processability and optical characteristics of the film) is attributable to the appear-

gin or a starting point for the thermal degradation of the PVC particles (Fig. 3).

The origins of black spots and past optimization measures are discussed in the following, together with a presentation of the current state of the art with regard to detection, evaluation and prevention.

Focus on Black Spots

Surface defects designated as black spots may range from very jagged with uneven geometries and definite black coloration to almost round, spherically shaped reddish-brown particles. The physical effects indicative of a defect are the different scattering behavior, less reflection compared to the surrounding surface and sometimes greatly reduced transparency. Depending on their origin, they may be distributed over the entire surface of the web, localized in a small spot or appear in great numbers in a smaller segment of the web.

<p>Operator error:</p> <ul style="list-style-type: none"> • Incorrect settings (v, T, t) • Wrong raw materials • Wrong formulation system • Inadequate cleaning • Over-/underfilling of the equipment 	<p>Process errors:</p> <ul style="list-style-type: none"> • Contaminants (hot trim, reclaim) • Temperature control (excessively long residence times & high temperature spikes) in the mixer and kneader • Overly high throughput • Formulation setting
<p>Material defect:</p> <ul style="list-style-type: none"> • Impurities • Lubricants, stabilizers have no effect • Thermal stability 	<p>Equipment error:</p> <ul style="list-style-type: none"> • Dead zones during material transport • Screw geometry • Surface finish (mixing blades, kneader screw) • Energy input (mixer/kneader) • Equipment condition (cleaning, technical defects)

Fig. 3. Overview of possible causes of black spots

ance of black spots or burned material particles. These are distributed randomly and irregularly throughout the web and have various sources. Black spots may originate in any individual system component from the mixer to the calender. Viewed on the basis of the temperature regime of the system component, a distinction can be drawn between locations that generate heat continually (temperature above 60°C), those with high temperature spikes or those with long residence times. These are generally the ori-

Detection and Specification of Black Spots

A visual inspection of the last few running meters of a mother roll is always performed by the responsible quality inspector or an illuminated exposed section of the moving web is inspected at intervals by the calendering crew during production. In this case, no accurate statement can be made about a distribution or exceeding a permissible number of defects. ▶



Fig. 2. Images of film defects (from left to right: speck, black spot, structure, hole, burn)

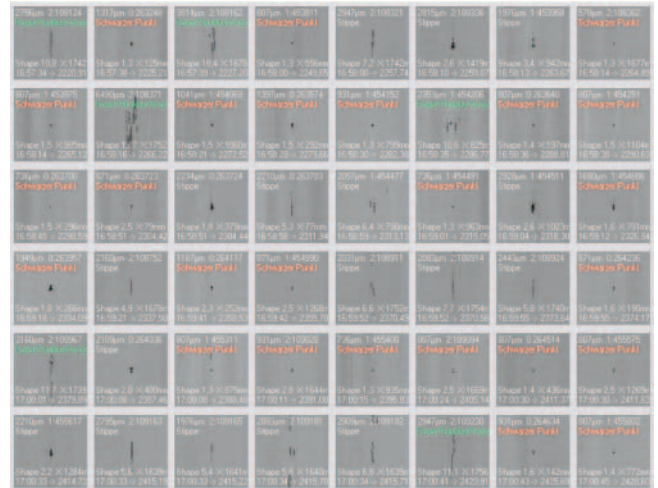


Fig. 4. Image of film surface (left), detected defects as logged (rights)

For a number of years, CCD cameras from OCS that inspect the entire surface of the web have been employed on all modern calendering lines (100 % inspection). Benefits include an objective evaluation of the entire production run as well as direct visualization of all defect types with marking as they occur (Fig. 4).

Customer-dependent specifications then follow from guidelines for pharmaceutical and food applications (Tab. 1). The comparison illustrates the greatly increased sensitivity and accuracy of the full-surface monitoring permitted by the camera systems using the same specification limit for visual inspection (per 10 m²) with, on average, a 200 times larger surface area for measurement and evaluation.

Processing Equipment as Origin

The temperature regime encountered during material flow defines the local temperature spikes in three pieces of equipment with the corresponding installation situations. These are the

- mixing section (with heated and cooled mixers),
- plasticating unit(s) (such as kneaders/extruders), and
- calender/calender gaps with auxiliary equipment (such as limit plates, cutter rolls or guard plates).

At these processing locations, the two primary variables – the elevated process temperatures and the increased residence time during material flow within this temperature spectrum – coincide (Fig. 5).

Mixers: A combination of heated and cooled mixers is used. Here, the formulation of the batch, which determines the calender throughput, forms the transition to production of a continuous web on the calender (Fig. 6).

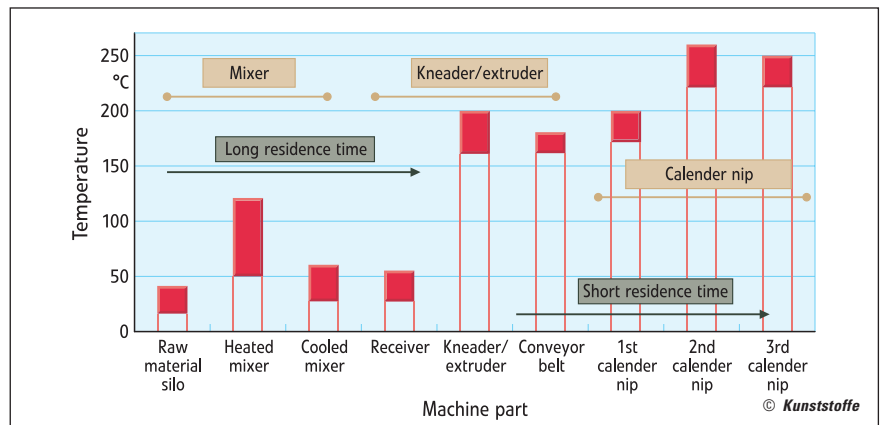


Fig. 5. Example of temperature ranges and residence times in the high-temperature process (4-roll L-calender)

In recent years, it has been possible to document the following causes for the formation of black spots in the mixing section:

- Adhesion of material in so-called dead zones (heated and cooled mixers),
- Speeds too high and mixing times too long,
- Surfaces of the mixing blades in addition to damage or wear,
- Material temperature at the discharge too high.

Geometry-related dead zones in equipment and exposed surfaces created by wear or chipping are the primary causes of the continuous formation of deposits and thus of black spots.

Kneaders / Extruders: The units being used from Buss AG, Pratteln/Switzerland, are co-kneaders from the PR 200 Series (L/D=11) with a degassing unit (vacuum degassing) and a rotating cutter to pelletize the material. The co-kneaders operate at speeds of up to about 300 rpm

	Visual inspection	Measurement system & evaluation
Film area evaluated	10 m ² (≈ 0,5 %)	Entire surface (100 %)
Equivalent defect area	Estimate	Calibrated measurement (resolution of CCD chip and v _{equipment})
Customer dependence	per QA code (customer)	Distinction as to application (Ph, FD, BC)
Evaluation aid	Eye / magnifying glass	Image of the defect with defect size
No. of allowed defects		
• Spots < 1 mm ²	1 [10 m ²]	1 [10 m ²]
• Spots > 1 mm ²	0 [10 m ²]	0 [10 m ²]

Tab. 1. Customer-dependent specifications

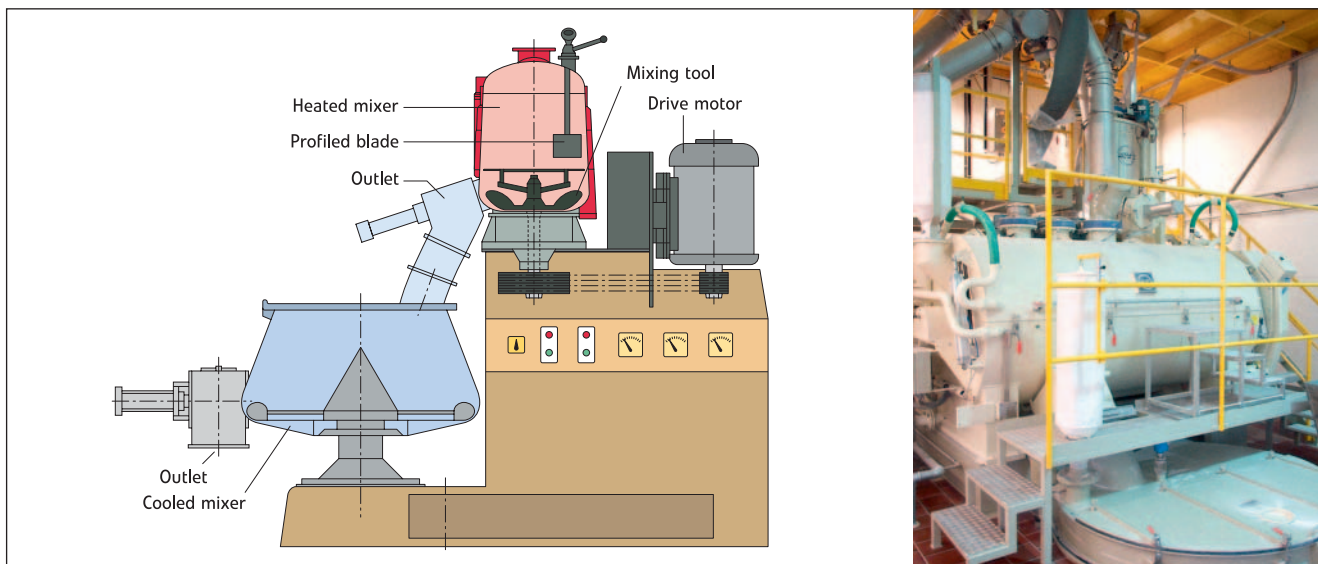


Fig. 6. Illustration of a heated and cooled mixer arrangement

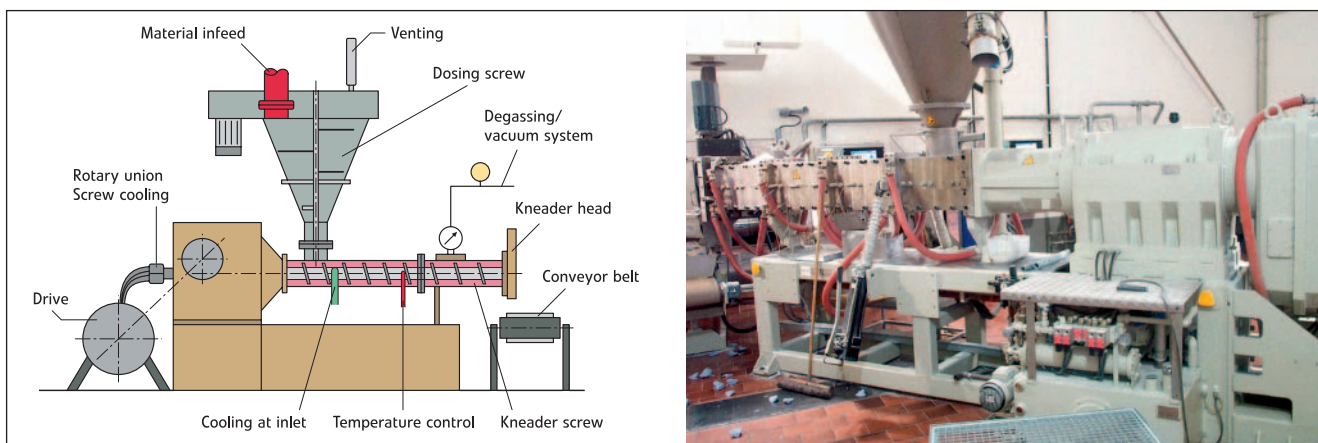


Fig. 7. Co-kneader for compounding of rigid PVC

– older equipment is thus sometimes operating outside the previously envisioned optimum range (Fig. 7).

The Buss co-kneaders that are used for the majority of work (and occasionally as planetary roller extruders in a bypass mode) exhibit the following problems:

- Accumulation of material in dead zones (geometry-based in spite of being self-cleaning because of incomplete meshing of flanks or defective surfaces),
- Energy input (temperature, speed) too high,
- Kneader blades (baked-on material outside the cutting surface or in the center),
- Inadequate/infrequent cleaning,
- Surface of housing and screw too rough,
- Poor degassing design – residue is retained,
- Unfavorable screw geometry and cooling,
- Material segregation in the receiving vessel/kneader hopper.

It has been further established that segregated and thermally degraded material tends to accumulate predominantly in four zones:

- The feed zone (region with the first few conveying elements),
- The transition zone (before and at the backup ring),
- The discharge zone (last few conveying element before the nozzle),
- The nozzle and the rotating kneader blade.

Calender: In the present analysis, the term “calender” applies only to the calender rolls 1–4 with all attachments such as limit plates or guide plates (Fig. 8 and title photo). Formation of black spots does not take place in any downstream sections of the line (take-off, subsequent processing), since the material structure of the film has already been homogenized and solidified. The following origins have been identified:

- Material accumulation on the limit plates at the 1st roll nip,
- Material accumulation on guard and guide plates (at the 1st calender nip),
- Cutter rolls (severely contaminated because of accumulated and burned material).

Raw Material as Origin

The purity of the raw materials on which the formulations are based is certified and monitored by the supplier. Impurities should thus seldom or never be the cause of black spots – but may be responsible in individual cases. The following factors are known:

- Thermal stability of the raw materials,
- Recipe of the formulation (amount of stabilizer, etc.),
- Moisture content of the starting materials.

Other factors are relatively inconsequential or cannot be confirmed using cur- ▶

rently available means of measurement or analysis.

Inspection Technology

In addition to providing a neutral evaluation of the film produced, the objective of all systems employed is direct marking of non-conforming regions of the web, 100 % inspection of a calendered film, retention of all quality data on behalf of the customer and visualization of system and process conditions for the operating staff in order to permit direct intervention to improve quality. The greatest results can be achieved here in the course of normal everyday production. Quality, which must be created or evaluated directly at the machine, can be improved considerably using such measurement systems and the amount of rejected material is minimized. If there is a tendency for specified product parameters to worsen, visual indicators are triggered. In this way, countermeasures can be initiated without delay or the sections of the web that would result in complaints from the customer can be removed in a subsequent operation and recycled to the material flow.

History / Development

Defect detection by means of CCD cameras began at Klöckner Pentaplast over 15 years ago. Use was restricted to calenders for extremely critical applications and selected customers. Five years later, the first trials took place at the technical center with various system suppliers. These lead to what has now become a 10-year relationship with OCS.

Development started with camera systems specifically in the area used for pharmaceutical films. In a joint effort, functions were implemented to help operators, engineers and quality manager use the system and the data obtained. In addition to continual refinement of the illumination technology, the actual operating software was expanded to include statistics and visualizations, the response speed of the cameras increased from year to year and the high-performance PCs employed permitted uncomplicated work with large amounts of data (Fig. 9).

Thanks to an "embedded PC" concept, today's advanced inspection systems offer a data rate of up to 160 MHz per camera. This allows storage and transmission of all measured data to a server via Ethernet. All data are analyzed in real time, and classified and evaluated on the basis of the customer's requirements.

Fig. 8. Conveyor belt for supplying material to a calender line



Depending on the application, CCD line cameras with 2,048, 4,096, 6,144 or 8,192 pixels can be employed. These achieve scan rates (imaging rates) of 72,000 per s at 2,048 pixels to 18,000 per s at 8,192 pixels. As a result, extremely high resolutions are attained in the lengthwise direction (direction of travel) even at very high web speeds. Through use of special objectives and the technological advancements in CCD sensors, the imaging quality of long CCD lines (e.g. 8,192 pixels) today is just as sensitive as that of short CCD lines (e.g. 2,048 pixels). For the film manufacturer, this means equally good inspection results for a considerably lower investment, since the price differences between the above-men-

tioned CCD line cameras are insignificant.

A simple example will serve to illustrate the benefits of using long CCD sensors: A rigid film calender with a width of 2,600 mm and a maximum web speed of 120 m/min is to be inspected at a resolution of 200 µm in both the lengthwise and crosswise directions. To achieve this resolution, either 4 cameras with 4,096 pixels each or 2 cameras with 8,192 pixels each can be used. This example demonstrates the better price/performance ratio of the approach using an 8,192 pixel camera as the result of lower procurement, maintenance and handling costs for equally good inspection results.

Depending on the configuration, the installed inspection systems send an alarm to the operator in the event of a critical individual defects (a black spot, a fly), defect trends (e.g. more than 10 specks with a diameter of 200–300 mm per m²) or if rolls or panels are outside of specifications. All data are stored and retained in the system. They are thus accessible at any time for comparison purposes or if there is a complaint. Production trends over longer periods of time, e.g. the last 12 or 24 hours, can be displayed online, and entire campaigns can be compared and analyzed offline in order to evaluate, for instance, raw material batches in relation to certain extruders in terms of the frequency of specks. Moreover, all systems provide printed logs of roll quality, matched to the needs of production and the customer.

Self-teaching Systems

Today's inspection systems come with an Easy Teach-In function. With the aid of this function, the operator can name defects on the basis of defect images and sort them by classes. The classifier automatically establishes the inspection parameters for individual defects. Since these systems operate with fuzzy algorithms, they are able to

! Company Profile

The **Klöckner Pentaplast Group** is the world's leading producer of packaging films for pharmaceutical and medical products, foodstuffs and electronic components as well as for general-purpose thermoforming, printing and special applications. Starting initially with one plant in Montabaur, Germany, Klöckner Pentaplast has grown in the mean time to 20 additional production facilities in 11 countries.

All pharmaceutical films produced are registered in the global DMF (Drug Master File) and comply with the relevant guidelines for pharmaceutical packaging such as FDA regulations, the BgVV and corresponding EU directives (Pharmacopeia). In addition, all facilities of the Klöckner Pentaplast Group are certified to DIN EN ISO 9001. All process sequences are monitored and controlled by a comprehensive quality management system. Production and hygiene standards for calendered pharmaceutical films are subject to the international requirements contained in the GMP guidelines of the World Health Organization (WHO).

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distinguish, for instance, flies from black spots. This is of considerable importance in the food industry, since the origin of the defect lies either in the cleanroom environment or in the production process. Furthermore, dust and lint attracted by static electricity can be recognized as such.

Illumination Technology

In principle, any light source can provide the illumination used as reflected or transmitted light. In most applications, an attempt is made to use transmitted light, since in this case both the upper and lower surfaces can be inspected. In contrast, physical irregularities such as scratches or extremely small air bubbles

hole during thermoforming and is thus absolutely critical. Because of its high opacity and speed, it is difficult to inspect the film using transmitted light; and inspection of both sides using reflected light is very expensive and requires a significant investment in technology. Moreover, black spots and specks located in the center of the film would not be detected. However, these could reduce the barrier characteristics of the pharmaceutical blister packs considerably after thermoforming because of thin spots or holes.

By using the right illumination unit, every black spot can also be detected on opaque materials, marked with a tag and removed during subsequent sizing to customer requirements.

port with written confirmation regarding calibration at the promised resolution (defect width), accuracy of the speed measurement (defect length, scan rate) and recording of physical dimensions.

This is followed by a check of the system using manually applied defined defects (e.g. black spots of various sizes) that the system should replicate in the same size.

At this point, the major step consists of validating and checking the accuracy of a collection of established, self-defined defects from production rolls. In addition, defects are classified, marked with tags and cut out separately. Here, the detected defect, the associated image with indication of size and the tag with description



▲ Fig. 9. Examples of integration of OCS camera systems in calender lines

in opaque films are detected reliably using reflected light. However, this requires very stable integration (stabilizing diverter roll) with very smooth movement of the web (Fig. 10).

Use of satisfactory illumination technology is of decisive importance for good and reliable operation of the camera systems. The standard approach employs high-frequency fluorescent tubes because of their low procurement and maintenance costs, but even fiber-optic and LED illumination techniques are used when needed.

An example of the successful use of fiber-optic illumination involves the inspection of covered pharmaceutical film at take-off speeds of up to 200 m/min (depending on thickness) and very high resolutions. Every black spot can produce a

Validation

The requirements and demands of the pharmaceutical industry are increasingly seeking to match detected defects with defined size and shape classes established in specifications.

The defects, which are usually described by the company, are assigned appropriately and managed by available systems. For the end user, the result is verifiable and documented certificates.

The procedure introduced at KP for such a verification chain (accuracy of the system, agreement of measured sizes and shapes, number of defects, etc.) begins with preparation of a customer specification for the system supplier as well as ordering/installing the system. The first important document is the acceptance re-

of the defect must all match. By measuring with a thread counter or microscope, the shape and size can be compared and evaluated.

Following discussion among the parties involved (production, customer, QC), specifications can be prepared and implemented, and production that is meaningful to the customer is assured.

The knowledge gained from recent work on customer validations confirms exact agreement between detected and tagged/removed defects. Validation binders in which the pertinent investigations are retained have been and will be compiled for appropriate types of film (pharmaceutical field – white opaque and transparent films).

OCS has also recently developed validation software for use with a transpar-



Fig. 10. Illumination concepts for detection of optical film defects



ent DIN A4 film that is placed on the moving web and is inspected as the web passes under the camera system at production speed. The validation film has four corner coordinates and within this rectangle very precise black dots in various sizes. The software scans the film in a separate mode as it passes, measures the image and compares the dots with the stored reference image. The resulting deviations are calculated for each defect dot and presented in the form of a report.

Comprehensive Quality Concept

Klöckner Pentaplast and OCS have worked together for years in the field of online quality assurance. In the mean time, almost all calender and extrusion lines as well as stretching frames have been equipped with inline inspection systems. Because of the high degree of integration, the inspection recipes are used worldwide once they have been prepared and guarantee the final customer the same high level of comparable quality from every production facility.

The systems are integrated into the company's intranet to a great extent and in the coming years will be fully linked to the existing production data management system. Each time a roll is changed, the measurement data for the particular roll are stored automatically on an internal data server with roll number and production date. As a consequence, all detailed roll data are accessible immediately for any future evaluations or in the event of isolated complaints (Fig. 11).

The lead operators use the system to start up and adjust the calenders. If critical specific defects or raw material and system problems occur, the operator is automatically alerted by means of a visual alarm and can take immediate action.

Every critical defect is marked with a tag automatically by the inspection system and documented in the accompanying printed logs.

Every five seconds, special software sends a screenshot of the current inspection system display to a computer in the quality assurance department. The software permits up to four defect panes (four

systems) to be displayed on one screen. The screen images give the quality assurance manager an up-to-date overview of all systems at any time. The same software is installed on the computers of the responsible manager, the laboratory and the application technician. In this way, the current quality can also be assessed at any time even away from the production systems.

Summary

Production systems are equipped as standard or are being retrofitted with the well-known components and the high functionality of the inspection systems described above. This assures comprehensive monitoring of the film quality produced with a similar data collection and retention structure at all facilities and on most machines.

The result is consistently high quality of all film produced and the assurance of continual optimization of the production process through direct intervention during production. The trend in the number of complaints as an indicator of a functioning quality assurance system shows definite improvement in recent years. If one considers the savings, to which the inspection systems have also contributed, a return on investment (ROI) of less than a few years can be assumed even in view of the high investment associated with the inspection systems. ■

THE AUTHORS

DIPL.-ING. ANDREAS SCHNABEL, born in 1970, works in the field of metrology and process technology at Klöckner Pentaplast GmbH & Co KG, Montabaur, Germany.

DIPL.-OEC. OLIVER HISSMANN, MBA (USA), is employed as Sales Manager at OCS GmbH, Witten, Germany.

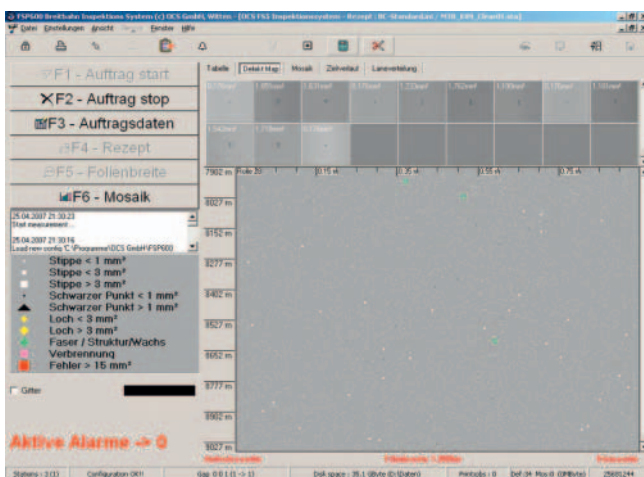


Fig. 11. Operator interface of the inspection systems

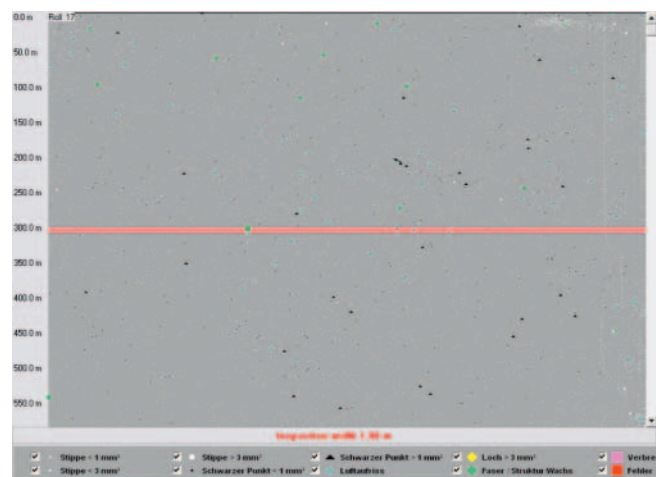


Fig. 12. Stored images from roll with alarms (tag)