9
FILMS, FOILS AND LAMINATIONS (COMBINATION MATERIALS)

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This chapter covers single layer, multi-layer and combination materials found as films, foils, laminations, coextrusions, coatings, etc.

Single ply materials

Although a proportion of 'flexibles' are multi-layer materials, a number of flexible packaging materials are found as single plies. Single ply materials are found in the form of paper, and those plastics which either do not require an additional coating to achieve a heat seal or can be employed as a direct wrap. Examples of these include various grades of polyethylene and plasticised PVC, which have to be high-frequency welded. A few foils may be used uncoated. (Note that most foils are varnished, lacquered or wash coated to improve scuff resistance or to assist print key.) Other single (monolayer) plastics may also be found as wraps which are restrained by a secondary feature (such as a tie or tape) or rely on special properties, e.g. cling films, for their retention around a product. Cling films, skin wraps, etc. involve such materials as thin gauges of plasticised PVC, Surlyn ionomer, modified grades of low-density polyethylene and Saran (PVdC) copolymers. Thus the use of single ply materials should not be ignored, particularly as these are frequently seen as being more environmentally friendly (i.e. multilayer materials are always more difficult to recover or recycle). Of the single ply materials listed above, paper needs special mention since it was one of the earliest wrapping materials and still has significant use worldwide.

Polyethylene, as LDPE, LLDPE or a mixture or blend involving combinations of LDPE, MDPE, HDPE, EVA, etc., finds a wide usage in bags, sacks, sachets, overwraps, shrink wraps, stretch wraps, etc. Most deep freeze packs, for example, use LDPE or an LDPE mixture which is produced from a reel on a form fill seal type machine. However, as many of these packs are up to 100% printed, even ink of 2–5 μm could be considered as a separate layer which modifies some of the physical and chemical properties. As all polyolefins need a surface (oxidative) treatment to ensure a good print key, this or any other surface treatment process may further modify the film properties.

Another use of single ply plastics is found in window cartons (cellulose acetate, polyester, regenerated cellulose, OPP, PS, etc.) and in plastic cartons (PVC). The use of thermoformings as bubble and blister packs for toiletries, pharmaceuticals, etc. and trays, soaps, etc. frequently falls in a grey area as to whether they are forms of flexible or rigid packaging. Frequently the final category is decided by the lidding material.

As mentioned earlier, foil can be found as a single layer material and applied as an overwrap using the dead fold characteristics of soft foil. Thin foil can be partly strengthened by embossing, but due to its extensibility it tends to demand an additional support ply.

Since it is sometimes difficult to identify whether a material is a single ply or a multiple ply, most students should examine a range of thinner materials to try to establish any constructional differences. However, many apparent single ply materials, e.g. biscuit, confectionery and chocolate (bar) wraps are actually multi-ply materials: these frequently use a type of OPP (oriented polypropylene—especially the pearlised variety) where a special (coextruded) outer and inner heat seal ply has been added. MAP and CAP food materials are likely to have an 'anti-mist' coating.

Finally, the use of recycled paper/board requires mention since continuous recycling leads to shortening of the fibres and a steady reduction in physical properties.

Although only a few single layer materials may appear to be used for pharmaceuticals, the largest use is likely to be found in shrink and stretch wrapping. As these may confer some barrier properties as well as acting as a means of collation and tamperevidence, they are expanded on below.

Shrink wrapping

If molecular orientation is introduced into a plastic, by extension under certain conditions, it will undergo deorientation or shrinkage, back to roughly its original dimensions, if subsequently reheated to the temperature above that at which it was earlier oriented. This property is normally achieved in a film by either the stenter process or the extrusion lay flat tubular process. The stenter process, which usually involves extrusion-casting, has one advantage in that the oriented film can be heat set (somewhat similar to annealing), thereby giving an improved degree of dimensional stability (reduces deorientation when heated). It is, therefore, more widely used for heat sealable wrapping films which are not used via a shrinking process. In both cases the orientation operation occurs at a temperature just below or above the softening point of the material in question. The most common shrink materials are low density polythene, polypropylene, polyvinyl chloride or ethylene vinyl acetate copolymers.

As the name implies, shrink wrapping utilises an oriented plastic which, when placed around an object, can be heated to a temperature where it returns, or tries to return, to its original dimensions—thereby forming a tight shrink wrap. Shrink wraps may be employed to wrap individual products, cartons, groups of cartons, packs in trays, etc. They can thereby be used to add to individual protection, act as a tamper-evident feature, improve certain barrier properties, act as a waterproof covering, or as a means of collating and protecting a number of items.

The shrink wrap material may be applied in two basic ways.

1 As a *full overwrap*—to provide a total wrap—where all of the item or items are enclosed by a film, i.e. an all-round wrap.
2 As a *sleeve overwrap*—a sleeve wrap where the ends of the longer direction are open and therefore exposed.

The shrink wrapping process involves several functions:

1 placing the film around the item(s) to be shrink wrapped
2 sealing the film
3 passing the unit through a heated (shrink) tunnel, arranged to give uniform heating, and avoiding hot and cold contact areas.

To create a total wrap, an L sealer or a fold-heat seal is usually required, while in the case of a sleeve wrap, a single longitudinal seal is used. The method of sealing is usually via a heated wire which may be used in conjunction with an impulse and pressure system or an impulse and radiant heat system.

While orientation gives a broad improvement in physical and chemical properties of plastics, i.e. improvements in clarity, tensile strength, inertness and reduced permeation to gases (oxygen, carbon dioxide), moisture, etc., these properties are lost once deorientation occurs.

Shrink films

Low-density polyethylene

Oriented LDPE is the most widely used of the shrink wrap materials. It has good strength, toughness and tear resistance. It also has good low-temperature resistance, hence is suitable for parts of the world with well below zero conditions. LDPE is a reasonable moisture barrier but is a relatively poor oxygen and carbon dioxide barrier.

Special grades of plastic need to be selected for satisfactory shrink films which need to cover a wide range of shrink ratios (machine versus cross direction). In the case of lay flat tubing, the ratio is achieved by bubble blow-up, rate of draw-off and other factors. Grade factors which are of special significance include density and melt flow index. Higher density materials have poorer puncture, impact and seal strength but have a higher shrink strength and lower percentage shrink. The melt flow index also relates to shrink strength, i.e. a lower MFI gives a higher shrink strength.

The incorporation of vinyl acetate into LDPE provides a softer material which shrinks at a lower temperature. Shrink temperature is around 108–115°C for LDPE.

Oriented polypropylene

This material offers high clarity, very good tensile strength and has high shrink energy. This means it should not be used on flimsy materials (e.g. light weight cartons) as this can give rise to distortion. OPP requires a higher temperature for orientation, hence needs more heat to give controlled shrinkage. However, PP has a much narrower deorientation range than LDPE and can prove more difficult to heat seal effectively. The shrink temperature is high, around 130–140°C.

Polyvinyl chloride

Both unplasticised and plasticised PVC shrink films are available. Plasticised material feels softer to touch, is less brittle, has a lower softening point and shrinkage. Unplasticised PVC has good surface gloss and clarity, but tends to be brittle, of poor impact strength and rather easy to tear once initiated. Shrink temperature is around 90–100°C.

Multi-layer materials

Multi-layer materials are used for special reasons, e.g. where an already shrink wrapped material has to undergo a further (secondary) shrink wrapping operation. This means that a different material is essential to prevent sticking, e.g. coextruded LDPE/EVA/PP where the PP inner layer will not stick to LDPE. Usage of multi-layer materials is limited.

Shrink wrap applications

Pallet shrink wrapping

This can be achieved using a preformed shroud (or hood), two single webs or a precut tubular form from a lay flat reel. In each case the perimeter should only exceed pallet size by around 7–12%. LDPE is the most widely used material, of 75 to 200 m gauge.

Transit wrapping

This may involve the collation of packages of similar or variable shapes. The most popular form uses either a direct shrink wrap (e.g. multi-packs) or a shallow tray which is usually made from corrugated board or solid board. Thermoformed plastic trays with a formed base (and/or a lid/cover) are also widely employed (e.g. bottles and aerosols). These systems offer restriction to pilferage and due to the see-through nature of the film may avoid the need for external labels. (Internal labels may be necessary if stock is controlled via a bar code.) Although LDPE has the major use, PVC may occasionally be used for its higher clarity. Gauges of 25 to 100 m are usually employed.

Display wrapping

Shrink wraps may be used on a number of materials (products) and packed items (e.g. cartons) to maintain cleanliness, to prevent or restrict pilferage or access to the product, to provide good surface gloss, etc. PP and PVC are used for their excellent clarity and sparkle. PP, however, has a narrower deorientation range and higher temperature than LDPE and PVC, hence may be more difficult to control. LDPE is available in highclarity grades and has a 'soft feel' (similar to plasticised PVC). Gauges of 25–40 μm are widely used.

In the above applications, either a sleeve wrap or a total wrap may be employed. The total overwrap has the advantage of excluding dirt and dust; it can also be a significant barrier to moisture. In the case of the total wrap, holes are initially required to allow any air to escape.

Reel materials

Reels may be unwound and used in different ways. L sealers usually use a single reel of a centre folded film.

In other systems two reels are used and these are welded together by a transverse sealing jaw. This is presented as a curtain to a moving product, so it is then enveloped by the curtain.

Shrink terminology

- Percentage free shrink—ratio measured in both the transverse (cross) and machine directions of the shrunk film versus its original dimensions.
- Shrink ratio—ratio of machine direction to cross direction shrinkage.
- Shrink energy—energy built into the film by orientation which is subsequently released during shrinkage.
- Retained shrink—shrink retained when film is tightly wrapped around an article (after shrinkage has occurred).
- Shrink strength—various oriented materials have different shrink energies, hence forces applied to the enveloped item vary.
- Film slip—this is the reciprocal of the coefficient of friction.

$$\text{Film yield} = \frac{\text{area of film (m}^2\text{)}}{\text{weight of film (g or kg)}}$$

Stretch wrapping

Stretch wrap films are elastic in nature, hence possess a memory. The film must have high elasticity, and once in position must not relax and lose tension (i.e. become loose).

Film materials

Linear low density polyethylene (LLDPE) is the predominant stretch wrap material, but films based on LDPE combinations, EVA and plasticised PVC are also found. Stretch materials can be produced by various processes, i.e. from lay flat tubing, casting, etc. The latter process gives orientation in the machine direction which assists stretch in the wrap around direction. Cling properties required for grip may be achieved by incorporating a cling additive or coextrusion to the outer surface(s) of a more tacky material, e.g. EVA.

Use of stretch films

Stretch-materials are usually applied in one of two ways. In the first of these, the material is extended by tightly wrapping it around an item or group of items in such a way that stretching occurs between the item and the unwound reel, i.e. it is extended at the point of application.

This process usually relies on the item or load rotating (e.g. a pallet) while the film reel is held vertically so that it can operate with a spiral motion. With simple hand wrapping, the operator walks around the item, applying layers which overlap in a spiral motion. Since these processes can only be applied to the sides of a pallet, a top sheet inserter may be employed to cover the top. This is then held in position by a stretch layer. The level of stretch achieved depends on the material employed, the uniformity of the load and the process employed. If a material is over-stretched i.e. the natural yield point is exceeded, the film becomes less puncture resistant and may break. Limits of stretch by these processes are around 50–60%.

Cling films usually can be categorised under stretch films.

In the second method, material is mechanically pre-stretched before application, using materials that can be stretched over 100%. This is partly because a pre-stretching process has a better control over the stretching operation, which occurs between two rollers placed near the reel of film. Since these two rollers are fitted with variable gears, the degree of stretch can be both controlled and altered. The width of the reel is maintained during this stretching process, which alters the properties of the film and increases the yield (lowers material used, hence reduces material cost). Improved methods of stretching are now available and can stretch the material three fold, i.e. up to 300%, by using motorised units.

Applications of stretch wrapping

Pallet stretch wrapping

This is the most popular use of stretch wrapping, hence it is frequently compared with shrink wrapping in terms of advantages and disadvantages. All the methods previously outlined are employed with LLDPE being the predominantly used material, in conjunction with a pre-stretching operation. Gauges of 15 to 50 μm are usually employed.

Stretch bundling

Stretch bundling can apply to single products or packs, or groups of somewhat irregular loads. The film is normally stretched around the ‘product’ to give a tight wrap with up to 25% stretch. The material finishes with an adhesive or heat seal. Modified LDPE, VLDPE or EVA are normally used.

Other applications for shrink and stretch materials

Skin packaging

Skin packaging employs material properties which are similar to stretch wrapping in that the film employed may stretch in the process. However, heat and vacuum are usually employed, hence stretch may also approach an orientation operation. In terms of material, Surlyn ionomer and Saran may be used as well as the more conventional materials.

Shrink sleeving

Shrink sleeving made from lay flat tubing or welded tubing is widely used as a tamperevident overwrap to enclose either a whole container or neck and closure. Materials include PVC, polypropylene and polyester. Shrink sleeving can improve container barrier properties or light exclusion.

Cling film

Cling films are usually elastic-type materials which will undergo stretch. The originally used cling films were based on plasticised PVC and PVdC (Saran) films. More recently, LLDPE and LDPE, particularly as mixtures with other plastics, are being widely employed.

Shrink versus stretch wrapping

Each of these has advantages and disadvantages which are given in Table 9.1. Further comparisons could be made against fibreboard outers which offer advantages in stacking strength and cushioning properties. This is often more important during transportation as in many warehouses stock is racked and not stacked two or more high. Both shrink and stretch wrapping offer significant cost savings over fibreboard if circumstances permit their use.

Table 9.1 Shrink versus stretch wrapping

Shrink	*Stretch*
Equipment more expensive, involves sealing, wrapping plus heat shrink tunnel.	Lower cost, frequently smaller.
More energy: orientation plus heat energy to shrink.	Less total energy stretching, no heat.
May not be suitable for heat sensitive items.	Can even be used under cold (refrigeration) conditions.
May distort under transit conditions.	Retains load more tightly.
Shrink in shrink wrap (two or more) may stick together.	Virtually no film-to-film sticking.
Shrink film will take up uneven contours more readily.	Will create areas of higher tension due to irregular products.
Needs different film widths for a range of sizes.	Needs fewer reel widths for a range of sizes.
Can be printed using distortion printing where shrinkage is uniform and well controlled.	Easier to print as stretch is mainly in one direction.
Can provide better weather protection etc., particularly if a total wrap.	Less protective, may not be totally waterproof.
Adds to general protection (climatic).	Lower climatic protection.
Uses (generally) more film (heavier gauges).	Uses less film.

Combination materials covering flexible and rigid applications (or multilayer materials)

The above heading has been selected to include all variants which may be now used for both flexible and rigid applications. Whereas previously these may have been introduced under ‘flexibles’, ‘film, foils and laminates’, etc., the use of new technology such as coextrusions, metallisation and different coating techniques has produced some confusion as to what terminology should be employed, so the word ‘multi-layer’ may be more appropriate.

General properties

Basically these combinations employ a range of materials to achieve certain specific functions, i.e. to offer:

1. support to other plies
2. barrier properties
3. heat sealing or sealability, including cold sealing
4. ease of decoration or printing
5. reflectivity when desirable
6. a means of bonding two or more materials together
7. ease of opening
8. security in the form of child-resistance, tamper-resistance or tamper-evidence
9. improved machine ability or improved machine performance
10. cushioning
11. acceptable cost
12. minimal use of resources (renewable or non-renewable)
13. suitability for disposal.

Certain materials, depending on caliper, grammage, grade, etc., may serve several functions. It is therefore only possible to indicate the primary function of some materials.

Base web materials

Base web materials may consist of or include:

1. paper
2. regenerated cellulose
3. plastic film
4. aluminium foil
5. coatings

- protective
- heat seal or cold seal
- surface appearance or texture (may also be protective)
- metallisation
- adhesive or tie
- waxes—surface or wax impregnation

6. plastic coextrusion and extrusion.

General construction—specifying

It should be noted that combination materials are specified (from the outside inwards) by weight (g/m^2), gauge or caliper. Most are made as a ‘reel’-fed operation and into ‘sheets’ if required.

Printing processes

Printing may occur before, during or after ‘combination’ and then the material is covered by a clear coating or a clear film. Although virtually any printing process could be employed, flexography and gravure are the main contenders for reel fed materials. More recently offset lithography has increased in use.

The base web materials and the role they may play are now covered in greater detail.

Paper

Paper may be made from mechanical or chemical pulp. The latter is preferred due to its purity, white colour (if bleached), strength, slow ageing, etc. Paper is usually below 300 μm in thickness; above this caliper the material falls into a 'board' or 'carton' category.

The advantages of a paper ply are as follows.

1 Basically of a non-toxic origin (be aware of 'dioxin')—starting with relatively pure cellulose fibres and water.
2 Relative low cost with ready availability. (Wood is preferred origin, but other sources are used.)
3 Available in a wide range of types, substances, finishes, etc., depending on fibre length and the method of finishing. Typical papers include:

- opaque glassine
- vegetable parchment
- MG sulphate
- tissue
- MG bleached Kraft
- super-calendered (SC) bleached Kraft
- coated papers
- glazed imitation parchment (GIP).

4 Various additives can be incorporated at the beating, refining or finishing stage, i.e. the paper can be modified to suit specific uses (improve opacity, printability, etc.).
5 Can be printed by a wide range of processes, especially prior to lamination.
6 Can readily be coated by emulsion, lacquer, solvent, or extrusion-based processes, etc.
7 Can be laminated to other materials with a low-cost aqueous adhesive due to its absorbent (porous) nature.
8 Has good rigidity and strength—hence makes a good supportive material.
9 Porosity can be adjusted to allow diffusion of gases, steam, etc. for sterilisation while maintaining a barrier to bacteria and moulds.
10 Opacity can be varied according to the fillers used. It can also be coloured or tinted.
11 Paper shows a degree of compression, hence acts as a slight 'cushion'.
12 Paper can readily be torn or cut open.

The main disadvantages of paper are as follows.

1 Paper is moisture sensitive and contains on average 7% moisture under 'normal' storage conditions. Changes in moisture may induce temporary 'curl' or distortion, or 'boil-off' when heated.
2 Paper has no moisture-resistant properties (unless specially treated), hence no barrier properties against moisture, gases, etc.
3 Has no heat seal or cold seal properties, hence special coatings or films are required for effective sealing.
4 Has poor transparency and gloss when compared with certain coatings and plastic films.
5 The porous nature of paper with air entrapped between the fibres acts as an insulator. This makes heat transfer more difficult, particularly on high-speed heat sealing equipment.
6 Paper is usually thicker than plastic films, hence gives less coverage for the same weight.
7 'Grain' in machine or cross direction confers various property changes.

Summary

Paper is widely used in laminations to give support and add strength; it is readily printed, easily opened, biodegradable, etc., all at a relatively economic cost. It is usually used within the range of 10 to 90 g/m^2. For further detail see Chapter 5.

Regenerated cellulose film (RCF) (Cellophane, Rayophane, Diophane, etc.)

The process of manufacture is detailed in Chapter 7. Natural RCF, apart from natural clarity, is similar to paper in its general properties, although it is a continuous film and not fibrous in nature, e.g. it is moisture sensitive and not heat sealable. Moisture permeability can be improved by coatings which can also confer heat sealability. These coatings, which may be applied to one or both sides, are usually based on nitrocellulose or Saran (PVdC).

These materials are covered by various earlier used codings:

- DMS—one side coated with nitrocellulose lacquer
- MSAT—two sides coated with nitrocellulose
- MXXT/S—solvent coating of PVdC, both sides
- MXXT/A—aqueous dispersion coating of PVdC, both sides
- MXDT—single side coated PVdC.

The coding definitions include:

- M, moisture vapour coating
- S, heat sealing
- D, one side coated
- X, PVdC coated
- F, extra flexible
- Q or P, high moisture permeability but heat sealable
- H, tropical
- QF, quick freeze
- PT or P, plain, transparent, colourless, non-moisture-proof
- C, coloured.

However coated, the base film remains moisture sensitive and gains or loses moisture via the raw (cut) edges. This means that the material shows a degree of dimensional instability and reels of material if stored under incorrect conditions will expand (dumb-bell reels) or contract at the edges. Most regenerated cellulose materials contain humectants and plasticisers aimed at resisting changes in moisture levels. Reels should, however, be stored flat and under controlled climatic conditions to avoid reel distortion affecting machine performance.

Summary

Although regenerated cellulose plants still exist, worldwide usage has significantly reduced due to the better dimensional stability properties of various polypropylene-based films. However, regenerated cellulose still finds usage for certain special applications, e.g. overwraps.

Cellulose derivatives are also found in other forms, as detailed below.

Cellulose acetate (e.g. Clarifoil)

Like regenerated cellulose this is a clear sparkling film with a high gas and moisture permeability (i.e. the film breathes), but has poor chemical resistance and is difficult to heat seal. Like RCF it has poor dimensional stability and has been used for window cartons and an external glossy film for laminations.

Films and coatings based on plastics

Films are continuous, thin, clear, coloured or opaque materials derived from organic polymers. Most polymers are synthesised whereas the 'cellulose'-based films mentioned previously are mainly of natural origin.

Films are usually less than 250 μm thick with most lying between 12 and 50 μm. Films can be produced from a number of processes:

- extrusion—from a slit die (flat die extrusion) or lay flat tubing (cylindrical die)
- extrusion plus calendering
- regeneration casting—as regenerated cellulose
- solvent casting
- calendering
- coextrusion—involving two or more materials.

Most of these processes are described in Chapter 7 and only exceptions are covered here, e.g. solvent casting involves a polymer dissolved in a solvent. The solvent is subsequently evaporated from the cast film by heat, thereby leaving a film. Cellulose acetate film is made by such a process.

Calendering is basically a rolling or roller process (like an old-fashioned mangle) where a plastic is heated and then rolled with heated and then chilled rollers. The caliper is controlled by the gap between the cylinders.

All manufacturing processes may be combined with other operations such as slitting, surface treatment (usually corona discharge), printing. Differing properties may be created in the plastic according to the conversion process.

Orientation

One special additional process is called orientation, where a plastic film is stretched at a temperature below its crystalline melting point in the machine or cross direction or both. The latter is called biaxial orientation. These orientation processes increase molecular alignment thereby improving strength in the direction of stretch, improving clarity, reducing permeation to moisture and gases and usually improving chemical resistance. When subsequently heated above the temperature of orientation, most oriented films will revert to their original dimension.

Oriented films are usually coated with a heat sealant which seals at a temperature below the orientation temperature, otherwise the film will shrink in the heat seal zone, crystallise and possibly become distorted (cockle) and brittle. Oriented films can be 'heat set' by special treatments.

General properties of films

Appearance

Films vary in transparency (haze); most are transparent with a glossy surface. Reverse side printing usually improves appearance and eliminates rub—but beware of process solvents affecting product. Films may also be tinted or pigmented (opaque).

Strength and flexibility

Most films are flexible and fairly strong. Some resist tear but usually tear easily once a tear point is initiated (slit, V-shaped notch, etc.). Most plastic films remain reasonably consistent for several years under reasonable storage conditions. Films generally age more rapidly when exposed and not in reels.

Heat seal

Most films can be sealed by some means—direct heat, hot air, ultrasonic, HF or RF, etc. Some need special heat seal coatings.

Protective properties

Generally all plastic films are water-repellent (water-resistant). Various degrees of barrier are offered against water vapour permeation, gases (organic and inorganic), oils, solvents, aromatics, preservatives, etc. All films, if free of pinholes, provide a barrier to moulds, bacteria, etc., i.e. are a hygiene barrier.

Permeation is usually measured as:

- gas: $cm^3/m^2/24$ h, at 25°C
- moisture—$g/m^2/24$ h, 90% RH 38°C (tropical) per 0.025 mm thickness –$g/m^2/24$ h, 75% RH 25°C (temperate).

The USA tests use 100 in^2 instead of m^2. The 'total' barrier depends on a number of factors such as caliper, area, gradient on either side of barrier, temperature and any damage due to creasing, printing, etc., including diffusion/solubility factors associated with permeant and film.

Films show different resistance to oil, solvents, perfumes, preservatives, acids, alkalis and other organic and inorganic substances.

Other special features

Other special features include shrink films, stretch films and cling films. These features are, however, unlikely to be used in combination materials although oriented plastics are used in cold formed blister packs.

Special individual films and their uses

All plastics are to some degree permeable to moisture, vapour and gases, but are considerably superior to untreated or uncoated paper. The following material factors may require consideration in the selection of a plastic film, but it should be noted that only some of the factors listed below are subsequently considered under the general review on each plastic:

- weight per unit area (g/m^2) and/or caliper
- yield and cost (depends on caliper/density)
- transparency—light transmission; haze
- tensile strength and tear resistance
- ageing characteristics—under light, oxygen, temperature, light and low-temperature performance, softening point/melting point
- water vapour permeability
- gas permeability—note that permeation of N_2, O_2, CO_2 is usually of a 1:4:20 ratio
- odour permeability (organic and inorganic)
- resistance to water, solvents, oils and fats, etc.
- ease of printing (choice of process/ink/need for pretreatment)
- ease of heat sealing (temperature range, dwell time, pressure)
- freedom from static or level of static
- odour and taste characteristics (food grade acceptance)
- adsorption/absorption of preservatives
- non-inflammable
- presence of additives, processing, aids, etc.
- slip characteristics/coefficient of friction (critical for form fill seal machines)
- toxicity (risks for food grade acceptance
- irritancy
- blocking—tendency for two layers of laminate to stick together
- dimensional stability—important with print registration
- stress crack resistance
- extractives
- melt flow index (MFI)
- converting characteristics—each process may modify the basic polymer in a small way e.g. sealability, by heat, high frequency, ultrasonic or impulse sealing; so they also need consideration.

The polyolefins

These include the

- polyethylenes (PE), density 0.90–0.96
- polypropylenes (PP), density 0.90–0.91.

Polyethylenes (polythenes) include materials designated as low, medium, high, and linear low: PE (LD, MD, HD, LL, VL, UL).

Low density (0.915–0.925) offers a reasonable barrier to moisture but is a poor gas barrier and odour barrier, is permeable to oil, perfumes, etc. and tends to absorb or adsorb certain preservatives. Some grades, particularly with a high MFI, are prone to environmental stress cracking (ESC) when under a stress (in-built or applied) and in contact with a stress cracking agent (e.g. detergents, wetting agents). LDPE may be modified by the addition of EVA which increases its flexibility (it is already a very flexible material), widens and lowers its heat sealing range and optimises heat sealing speed. (LLDPE is steadily increasing in use and can generally be used where LDPE is referred to.)

As density increases (MD 0.925–0.935, HD 0.935–0.965), the material increases in rigidity, improves in barrier and chemical properties, becomes more difficult to heat seal (higher temperature), reduces in transparency and increases in haze.

HDPE is approximately a threefold better moisture barrier than LDPE of equivalent thickness. Polythene surfaces need pretreatment prior to printing or adhesive lamination.

Uses for the films include:

- heat seal inner ply in laminations, sacks, shrink and stretch wrapping, etc.
- LDPE is frequently used as a lamination ply to bond two materials together
- HDPE is employed in boil-in-the-bag applications.
- All have applications as bags.

LLDPE is actually a copolymer of ethylene, with butene, octene or hexene. It is finding increasing use due to economies of polymerisation and film strength. It provides the strongest heat seal of the PEs, has more extensibility and a capability of being downgauged. Other ethylene polymers include the following.

Ionomer (Surlyn) is a methacrylic acid and ethylene modified molecule with a metal ion (sodium, zinc, magnesium). Easy sealing, soft, strong, grease-resistant clear film, and seals well in contact with contaminants. Puncture-resistant, with a high hot tack. Approximately double the price of LDPE, but can be used in thinner gauges. Can be used in the inner ply of laminates, at approx. gauge of LDPE and as a skin pack over sharp or pointed objects.

Other ethylene copolymers

EMA, ethyl methyl acrylate, is a random copolymer consisting of a polyethylene main chain with methyl acrylate side branches. One main application is the film used for surgical gloves. It is more flexible than LDPE, less crystalline and much softer with a rubbery elasticity. It also has low softening and heat seal temperatures, good strength but poor optical properties. It can accept high pigment and additive loadings and is therefore widely used as a carrier for masterbatches.

Cast polypropylene (PP) is extensible, moisture-resistant, clear and seals at temperatures above those used in steam sterilisation. Oriented PP is strong and can be used in thin gauges. Unless coated or coextruded the film cannot be heat sealed without distortion. OPP is very clear, but can also make an opalescent film. Has a similar degree of inertness to HDPE.

Unless PVdC coated or metallised, OPP is a poor gas barrier and needs pretreatment prior to printing.

Oriented PP is used for overwrapping cartons, generally as a replacement for regenerated cellulose. Cast PP is used as the seal layer for packs designed to withstand autoclaving. Special grades of PP are now available for blister packing. Woven PP with across and diagonal plies are used for sacks and bags. Trade names: Propophane, Propofilm.

Polyvinyl chloride (PVC, density 1.35–140)

Clear, good gas barrier, does not heat seal. Found in the following forms.

- Plasticised PVC—rarely used except as bags (IV solutions, blood) or as pillow packs. Sealed with special adhesives, or by HF/RF welding. Plasticised film is highly moisture permeable.
- UPVC (unplasticized PVC)—may contain a low level of modifier (vinyl acetate). Has a low permeability to oxygen but is moderately permeable to moisture. The material is fairly rigid and is not heat sealable.

PVC is used in overwrap film, shrink film, shrink sleeving, thermoforming for all types of blister and bubble packs. Plasticised PVC IV bags are usually PP/Nylon overwrapped to reduce moisture loss.

Polyvinylidene chloride (Saran, PVdC, density 1.65–1.70)

PVdC is a soft cling type film but very strong. Difficult to handle. An excellent barrier to moisture, gases, grease and odours generally. Has a fairly narrow heat sealing range unless modified. May discolour slightly and embrittle slightly with age.

PVdC has limited use as a film but may be used as a central core in some coextrusions. Often used as a coating material, e.g. on other films as a good barrier and heat seal.

Polystyrene (PS, density 1.05)

Relatively highly permeable to moisture, and fairly permeable to gases. Used for thermoforming (non-barrier usage) and as a shrinkable film on a limited basis. Relatively brittle material unless impact-modified when clarity reduces.

Used mainly in blister-type packs (and as bubbles on cards for display) and OPS for some labels.

Fluorochloroethylenes

Chlorinated and particularly fluorinated derivatives of ethylene usually offer high inertness and good barrier properties. One film, Aclar (trade name) based on polymonochlorotrifluoroethylene (PCTFE), is the most moisture-impermeable commercial film currently known. For a similar thickness it is approximately ten times less permeable than PVdC. Derivatives generally have high melting and softening points. Found both as homopolymers and copolymers. For detail see Chapter 13.

Polytetrafluorethylene (PTFE, trade name Teflon)

PTFE is a very hard, chemically inert, low-friction material. It is mainly used to coat machine parts (to reduce friction) and heat sealing jaws to aid clean release. It is not used as a lamination film but has been used as a coating on closures (densities up to 2.2).

Polyvinylidene fluoride (PVdF)

More inert than PVdC; may find some application, but is more expensive.

Polyvinyl fluoride (PVF)

Has high weather resistance, but again, high costs restrict packaging applications.

Polyester (polyethylene terephthalate, PET; density around 1.38)

Usually found as a cast film. It can readily be oriented and heat set. It can be produced in thin gauges (down to 12 μm) as it is an extremely strong and tough material. Polyester is also clear and glossy.

It is heat seal resistant unless produced as a coextrusion or coated. A good barrier to most gases and volatiles, but only fair to moisture. It is easily metallised. Found as PETP and PETG (Kodak), Pet G contains an additional glycol molecule.

Often used as an outer ply to laminates and makes a good abuse-resistant layer. Use of metallised polyester is increasing as single and double metallised layers. Difficult to tear—needs a tear propagation point.

Thicker grades can be thermoformed (medical and pharmaceutical applications) and subsequently sterilised by steam autoclaving, gamma irradiation, etc. Non-oriented PET has a high melting point, around 250°C.

Polyamide (Nylons, density around 1.1)

Although various grades are available, Nylon 6 is mainly used for films (also 6:6 and 11). Generally flexible and tough at low and high temperatures. Only fair in resistance to grease and oils, with low gas permeability and a poor moisture barrier (due to absorption of water). Can be heat sealed in spite of high temperature and steam autoclaved. It can also be thermoformed. Usually slightly hazy although oriented nylon is clear. Usual gauges 25, 50 μm with orientation producing films down to 12 μm.

Combined with polythene to give various thermoformed packs for cheese, bacon, etc. Also used as an outer ply for boil-in-bag applications and as an outer ply in some cold formed blisters.

Polyvinyl alcohol (density 1.25)

PVOH is a water soluble film, with good gas, odour and grease barrier. Used as a water soluble sachet, but usually needs protection from moisture.

Polycarbonate (PC)

Another tough, clear film. Withstands steam sterilisation. Rather expensive. Only fair moisture resistance but good scuff resistance. Used in some laminations.

Polyacrylonitrile (PAN, trade name—Barex)

Good gas barrier. Only fair moisture barrier. Clear but not as clear as some other films, i.e. polystyrene, polycarbonate, polyester.

Polyurethane (PU)

Strong and rubbery. Frequently used as an adhesive or tie layer. Widely used as foams.

Ethylene vinyl alcohol (EVOH, trade name—Eval)

Good gas and odour barrier. Good moisture value when dry, but as moisture is absorbed, moisture barrier properties reduce. Relatively expensive. Usually used as a central ply in coextrusion processes. Replacing foil layer in some laminated tubes. Good barrier to certain flavours: peppermint, spearmint, etc.

Spun bonded materials (Tyvek)

Very tough with paper-like appearance. Used in medical packaging as a steam sterilisable porous material, (excludes bioburden). Based on HDPE. Has high strength.

Pliofilm—rubber hydrochloride

Was an excellent sealing medium but had few other properties to recommend it. Still available on a limited basis. Deteriorates rather rapidly with age.

Coatings

Coatings are an alternative means of adding properties which are not present in the base material: improved barrier, heat or cold sealing, improved appearance, adhesion, etc.

Types of coating processes

- Water-based—usually as a dispersion or emulsion coating, e.g. PVdC, cold seals.
- Solvent-based—followed by evaporation, e.g. heat seal lacquers, high-gloss lacquers, primers and key-coats.
- Vapour—e.g. vacuum metallisation.
- Molten materials—are applied hot then allowed to set, e.g. waxes, hot melts, plus extrusions or coextrusions and such new coating processes as plasma enhancement, sputtering, vacuum deposition, etc. (see below).

The application of coatings

Other than the newer coating methods mentioned above, many coating methods apply a liquid-based material to a solid web. These coatings may be applied either as a continuous (overall coating) or by a pattern system which may involve a ‘printing plate’ principle. Coating processes may apply excess, followed by controlled removal of the excess (e.g. by a ‘doctor’ blade system) or by a controlled (premetered) amount being applied directly (Figure 9.1)

Deposition can also be obtained by electrostatic spray or electrodeposition.

Typical examples of coatings are as follows:

1. Nitrocellulose, which was one of the earliest coatings used, as per MS regenerated cellulose film.
2. Saran or PVdC (usually a copolymer of vinyl chloride and vinylidene chloride). Applied by either solvent or aqueous dispersion coatings, with high coating weights (up to 180 g/m^2) requiring a number of coatings. Dispersion coating generally tends to have better moisture barriers.

PVdC may be used as an internal barrier-sealing ply or an external layer for protection and gloss.

Lacquers and waxes

1. Widely used as an external coating to provide a protective coating to the print, and to provide a product-resistant finish. UV cured lacquers, varnishes and inks can offer a very high gloss.
2. Microcrystalline waxes may be used for barrier properties or as a heat seal. They may be used as either a surface layer or as a total impregnation (paper). Known as wet and dry waxing respectively.

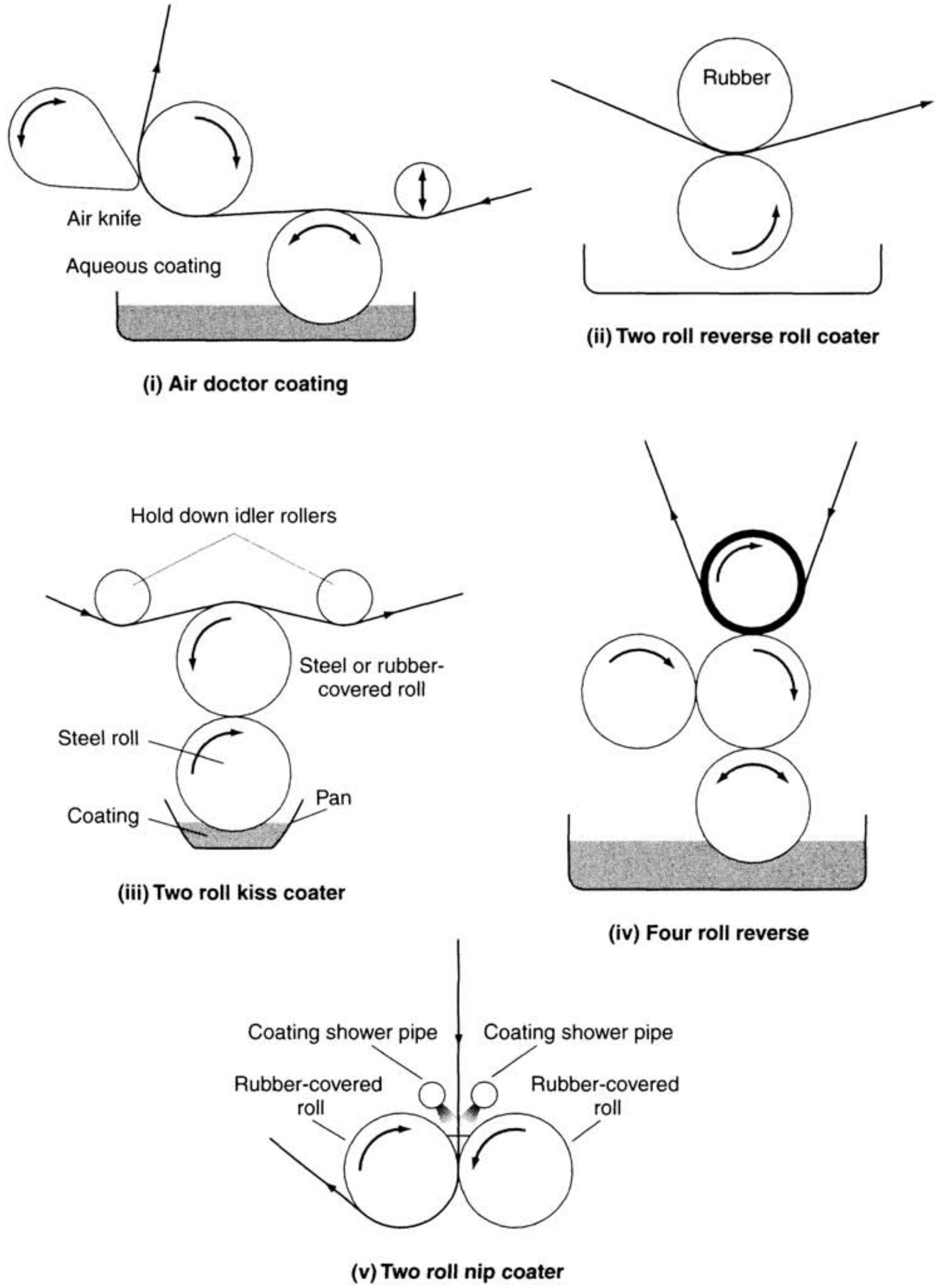


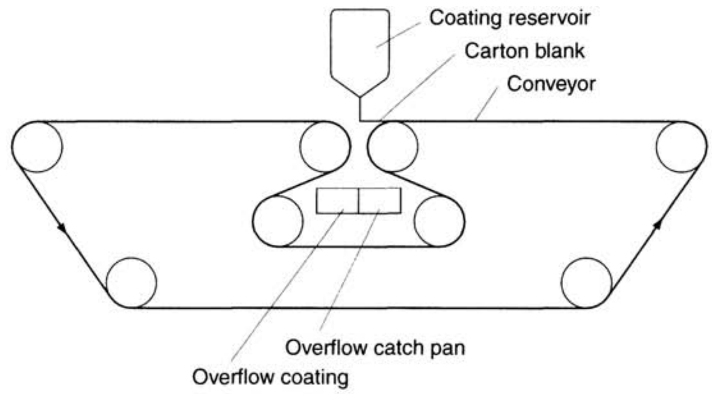
Figure 9.1 Liquid coating processes: (i) excess application technique; (ii–viii) predetermined (measured) systems; (ix) for higher viscosity materials

3 Hot melts and adhesives. May be used in a lamination process, but are usually considered as coatings. Hot melts may confer some protective properties, even when used for adhesion purposes, as they are based on plastics, i.e. are 100% solids.

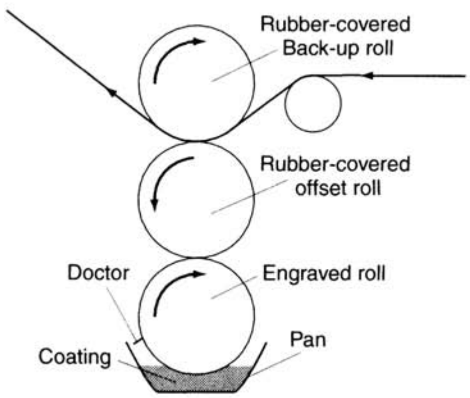
Extrusion coatings

Extrusion is a process where plastic film is delivered hot onto a substrate ply. A more economical way of attaching a ply of plastic and widely used for LDPE. Is less practical with certain other plastics due to risk of degradation (PVC) or the high temperatures employed. Direct extrusion and coextrusion enables use of thinner gauges of materials typically down to 12 μm.

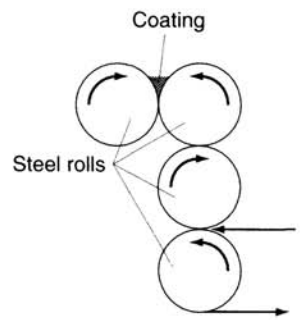
Coextrusion coatings usually employ materials of similar viscosity, softening or melting points, whereby two or more layers can be joined together internally or externally by extrusion dies.



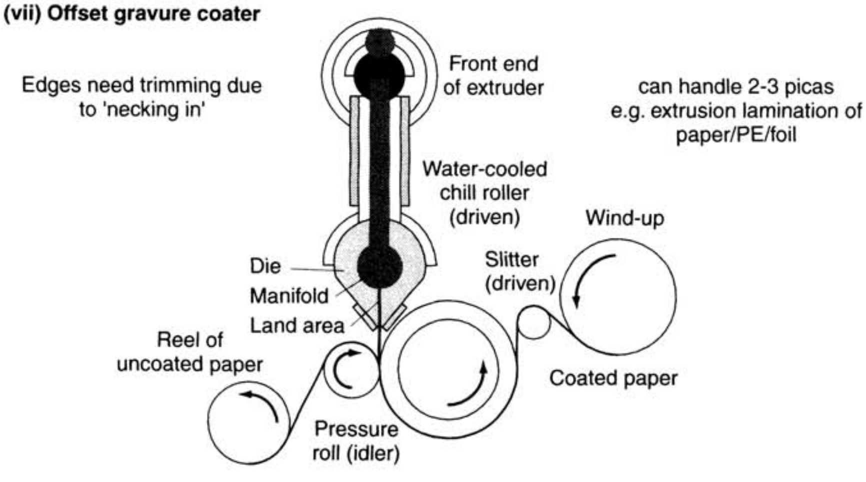
(vi) Curtain coater



(vii) Offset gravure coater



(viii) Four roll calender coater



(ix) Extrusion coating paper

Vacuumised treatment, e.g. metallisation

Used to deposit a coating of aluminium onto the surface of films. Gives foil type appearance, reflecting light and heat and reducing moisture and gas permeation. Covering of particles is not continuous and can be demonstrated by holding up to light when the degree of visibility enables items to be identified through the film. Using two metallised webs laminated with metallised area to metallised area contact gives much superior protection. Lamination to a flexible substrate reduces cracking of the metal layer which can happen when the material is creased or flexed, i.e. by the addition of an LDPE or LLDPE layer to give PET/metallised/LDPE.

FoilÐaluminium

Aluminium is the most abundant metal on the earth's surface, but it is one of the most costly constituents in a laminate.

Foil is obtained from metal of 99% purity and above. The gauges range from 0.006 mm to 0.040 mm. The foil is annealed to give a soft foil with a 'dead fold' property. Hard tempered (non-annealed) foil occasionally finds special applications, i.e. push-through lidding for blister packs. Lubricants are removed from hard foil by either solvent washing or controlled heating. For any nominal gauge +8% variation is normally allowed.

Foil offers the following properties:

- attractive metallic appearance
- brightness and reflectivity (light and heat)
- no light transmission; total barrier
- odourless, tasteless, non-toxic
- hygienic (process of manufacture eliminates any microbiological contamination; it will not support growth of bacteria or mould)
- excellent barrier to moisture and gases—even pinholed varieties offer better protection than plastics and papers (particularly when laminated to a plastic ply).
- can be printed and embossed.

The disadvantages of foil are that:

- it is not heat sealable
- in thin gauges it is extensible, hence needs a support layer
- under unfavourable conditions foil may corrode, either in contact with 'chemicals' or due to bimetallic corrosion (e.g. in contact with a ferrous metal)
- surface oxidation causes loss of lustre
- perforates or pinholes relatively easily with thin grades, when creased, folded or excessively flexed.

Where thin foils are used in laminations the bright or dull side needs to be nominated. Matt usually gives the best adhesion. This may also be improved by the use of a key or primer coating, also called wash coatings, weight 0.5 to 2.0 g/m^2.

Pinholes and moisture permeation

Foil of 0.038 mm is guaranteed pinhole-free; 0.017 mm can be considered commercially free for most purposes. Lower gauges gradually increase in the likely number of pinholes, e.g. 0.009 mm foil may contain 100–700 pinholes per m^2 and 0.012 mm foil 30–150 per m^2. If these are laminated to plastics then the permeability relates to the area of pinholes and type of plastic when equated against the total area of foil. Hence the permeation figures remain low unless the perforations are increased or enlarged during the lamination and subsequent machining operations on the packaging equipment. For this reason pick-up on finished packs is more relevant than direct permeation figures on the basic packaging material. Gas permeation is usually greater than moisture, hence could be more critical.

Foil strength

Soft aluminium foil has a fairly low tensile and tear strength, so it is essential that very thin gauges are supported by paper or film. In general, foil of 0.025 mm and below has to be supported. For example, 0.025 mm foil laminated to 30 g/m^2 LDPE is a widely used strip packaging laminate. In theory, 0.015 mm foil laminated to 30 g/m^2 LDPE would be a more economical proposition. However, at this caliper any undue stretch would be likely to perforate and tear the foil. Hence it is possible to reduce the foil caliper only if support is increased, i.e. by the addition of a paper ply. Thus if cost savings are to be made the final laminate would probably be either

- 37 g/m^2 GIP/0.012 mm foil/30 g/m^2 PE, or
- 44 g/m^2 GIP/0.008 mm foil/23 g/m^2 PE.

Foils used on the external ply are invariably given a light coat of lacquer to reduce scuff and improve slip. However, if paper is a middle ply any excess moisture may literally 'boil off' during the heat sealing operation. Paper as an external ply is therefore generally preferred.

Foils of 0.006 mm and 0.007 mm are now offered and may be incorporated into a variety of laminations. Special thicker foils of 0.040mm (40 μm) and above (up to 60 μm) are used for some cold forming operations.

Summary

Flexible packaging foil is usually coated with heat sealing material or laminated to other plies that include a heat sealable layer. Lacquered, sometimes embossed, foil is used to lid containers, e.g. blister packs. Foil is used in a wide variety of laminates. However, it cannot be used in any coextrusions, hence alternative barrier materials have to be used.

Laminations and lamination processes

Laminates are combinations of various plies created to obtain the properties which are not provided by one material alone. They use the minimum of materials (thin gauges and low grammage), and are cost-effective. However, they conflict with certain environmental issues, as recycling and/or reuse is either impossible or difficult.

Two widely used laminations are paper/foil/polythene and paper/foil/Surlyn. In these constructions the paper provides strength, brilliance and printability, the foil provides an excellent barrier to moisture, oxygen, light, odour and flavour (loss or gain), and the polythene or Surlyn gives heat sealability. These are widely used for strip and sachet packaging.

Lamination processes

Lamination may be achieved by adhesives, extrusion and coextrusion (Figures 9.2 and 9.3).

The more traditional method to make laminates uses separate plies combined with adhesives, which can be divided into groups—molten, water-based and solvent-based. Wax and polythene extrusion are the main molten laminants. Water-based glues are often used to combine paper and foil. Solvent-based adhesives include the polyurethanes, but recent developments use water dispersions and molten curing systems to replace the solvent systems. Cross-linking reactions develop high heat and product resistance in all these adhesives.

Extrusion consists of extruding a film from a slit die where the hot film may be nipped in contact with a second material and then cooled to give a bond between the two (or three) materials. Extruded LDPE is widely used both as a film in its own right and as a ply which combines plies on either side of it. Circular die extrusion can also be employed without the 'nip' stage in slit die laminating, i.e. it involves true coextrusion.

Coextrusion produces a multi-ply material directly from the individual resins. The method is limited to thermoplastic materials such as polythene, polypropylene and nylons. Thin layers of extruded bonding resins are necessary to combine many of the resins. Coextrudates have to be surface printed and the outer film cannot be reverse printed as it is often used with more conventional laminates with a film outer ply.

Adhesives—traditional

Various types of adhesive may be employed, i.e.

1 aqueous—with paper-based materials or where moisture can be lost from or through one web
2 solvent—loss of a volatile carrier by heating and special ducting
3 hot melts—advantage of virtually instantaneous set once heat is removed
4 hot wax—similar to hot melt except that wax remains pliable for a longer period.

(1) and (2) are termed 'wet' bonding while (3) and (4) are known as dry bonding processes. However, the second type (solvent) can also be used as a dry bond, where the solvent adhesive is applied to the substrate, excess solvent is evaporated off and then the second ply is bonded to the tacky adhesive via a nip roller.

Adhesive coatings may be applied by several means, i.e.

- excess of coating is added to the web and the surplus is removed by means of a rigid knife, flexible blade, or air jet knife, i.e. 'doctor' systems
- a controlled amount is applied to the web by means of rollers (roller coating), brushes, a calender, or by curtain coating.

A coating may be achieved by passing the ply between a coating roller and a back-up roll (usually rubber) or by kiss coating whereby the tensioned web makes contact with a coating roller. With certain more difficult materials a nip roll system (hot or

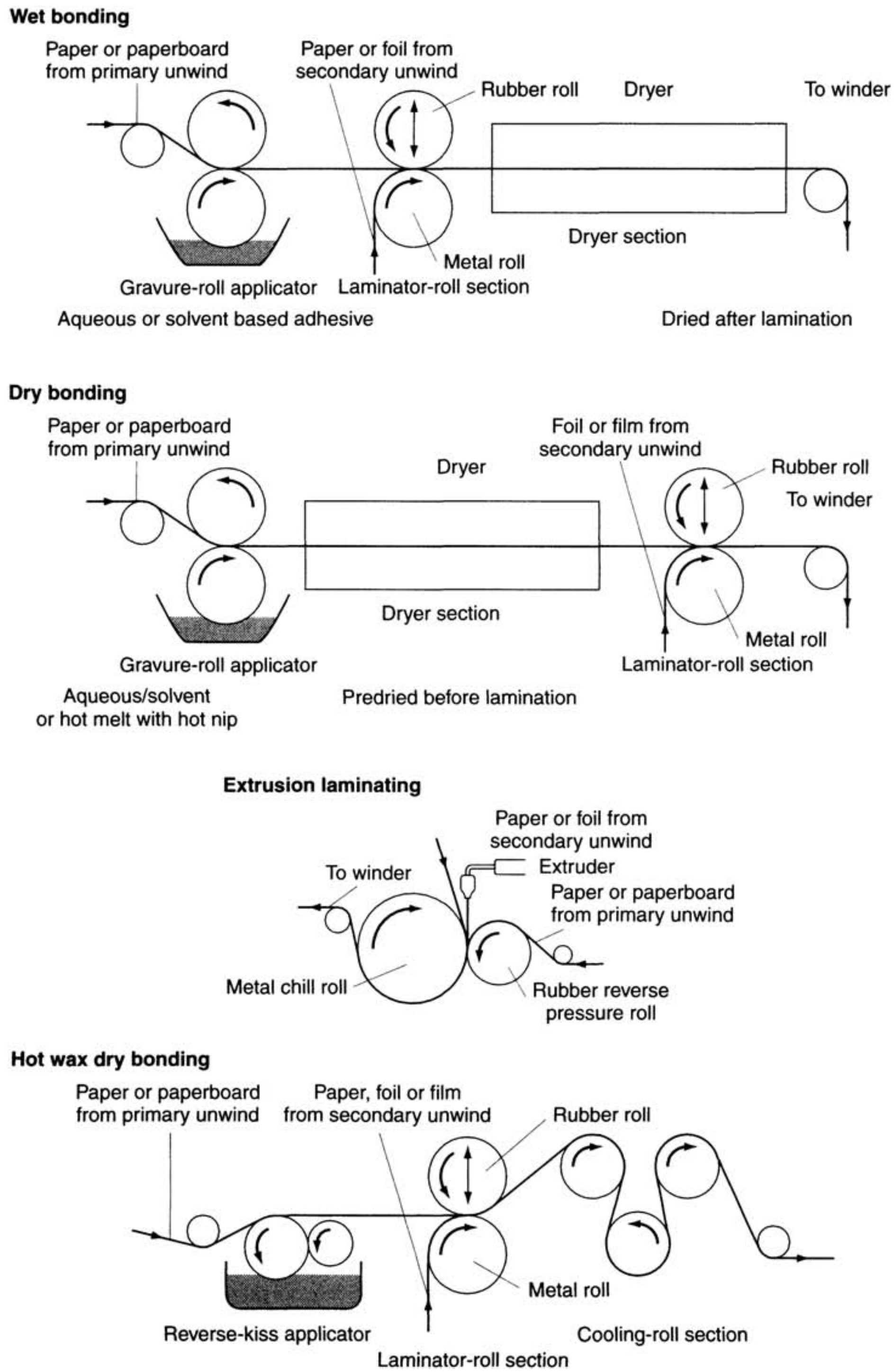


Figure 9.2 Lamination: wet and dry bonding

cold) may be employed. In this instance the area between web and roller is flooded with the coating medium. Gravure cylinders may also be used for coating.

In general, hot melts offer production advantages in cleanliness of operation and the elimination of solvent extraction problems. Hot melts can also be modified to contribute to the barrier properties of the laminate. Control of heat and cost are the main disadvantageous factors.

More modern systems include polyurethane and acrylic-based adhesives and twopart curing adhesives are gaining popularity.

Examples of the various laminations used include:

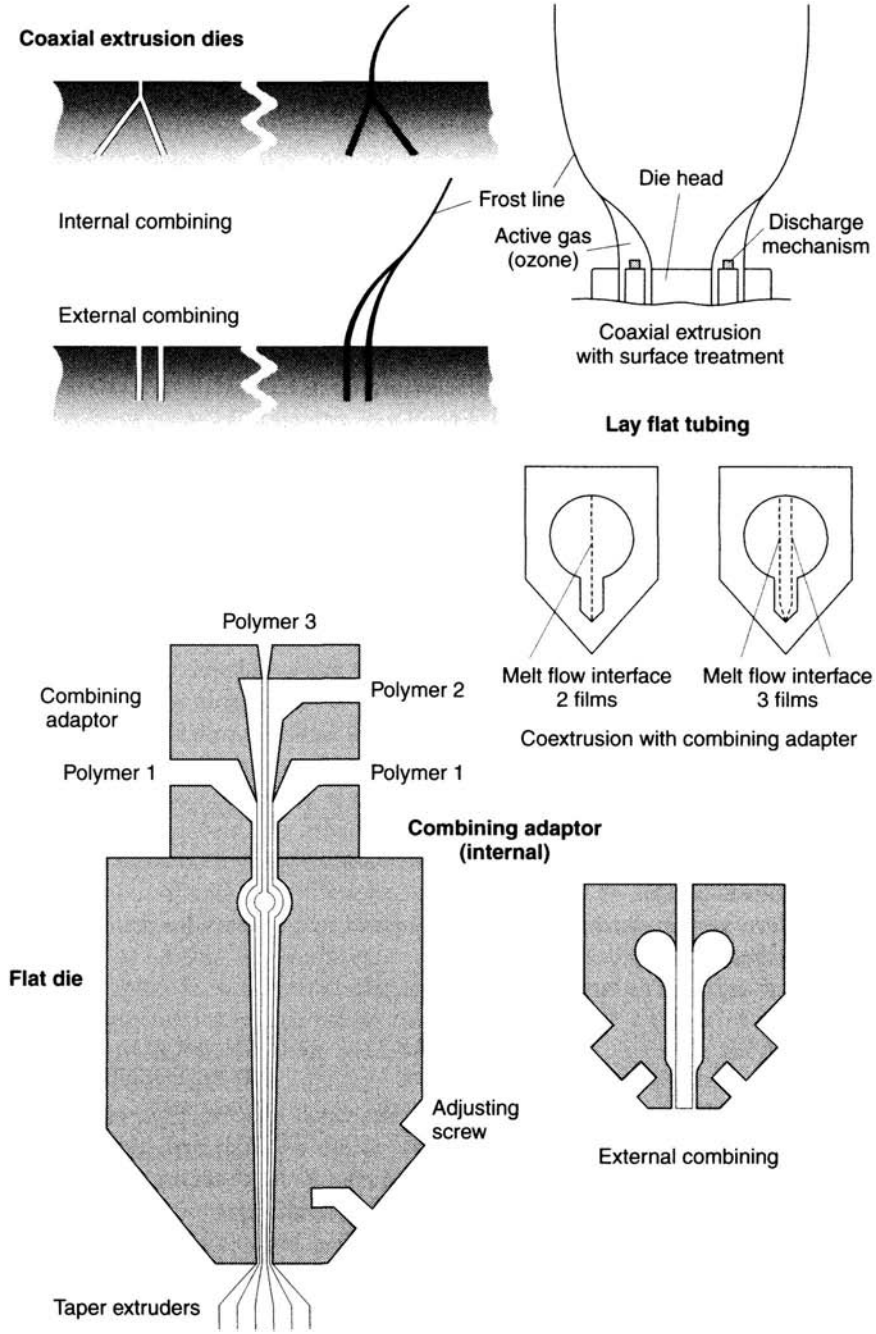


Figure 9.3 Extrusion and coextrusion processes

1	OUTER PLY	CENTRE PLY	INNER PLY
	PAPER (strength, appearance)	FOIL (barrier)	POLYTHENE OR SURLYN (heat seal)
2	OUTER LAYER	CORE	INNER LAYER
	COPOLYMER (heat seal)	POLYPROPYLENE (strength/barrier)	COPOLYMER (heat seal)

Laminate 2 has the advantage that it will seal to either the external or internal layer to itself, i.e. an overlap seal can be achieved.

Coextrusion is the latest process by which a series of plies can be joined together in the hot state. This may involve direct adhesion between plies or the use of extruded 'tie' or 'adhesive' layers. These bonding layers are frequently only a few micrometres thick and involve relatively low grammage levels. However, coextrusion is only cost-effective as a long-run process since set-up times can be lengthy.

Table 9.2 Provides a comparison between laminates and coextrudates

Laminates	*Coextrudates*
Versatile, include thermoplastic, paper and foil.	Limited to thermoplastic materials of similar viscosity. Exclude paper, foil, metallisation.
Sandwich print and coating can be used for good appearance and protection.	Surface print and coating only can be used.
Various adhesives can be employed. Crosslinking adhesives are often used, giving good heat and product resistance. Older adhesive systems are more prone to delamination.	Thermoplastic adhesives have to be used with limited product resistance. Some layer combinations do not require adhesives or tie layers.
More rigid.	Less rigid (especially cast).
Extra cost is necessary to pre-make individual layers before laminating. These layers also have a certain minimum thickness (limitations related to producing and handling this material).	Most cost-effective, especially for long runs. Thinner layers of more expensive materials can often be used. Setting-up operation needs greater expertise and may be lengthy, with various levels of wastage.

Decoration and printing

Materials may be surface printed, reverse side printed (if transparent) or sandwich printed (between two plies—either surface or reverse side). Two basic printing processes are usually employed:

Flexography and gravure (offset lithography may eventually come on stream).

Flexographic and gravure or photogravure printing

Flexography was a relief process on a rubber or composition type stereo mounted on a printing cylinder. Originally suffered from squash-out but better control on registration means that good half tone printing can now be achieved.

Gravure printing employs an intaglio process whereby the print area lies below the surface of the plate in small cells. The tone or colour depth may rely on the depth of etch (i.e. amount of ink which lies in the cells) and/or the number of cells per linear inch. All gravure is basically half tone (solid line gravure plates are used in some tampon or cliché processes).

Gravure printing plates are expensive, £750–1,000 per cylinder (per colour), so four colour printing will involve an outlay an of £3,000–4,000. Make ready is also fairly lengthy, hence long runs are necessary to offset the high cost of the setting-up. Gravure gives very high-quality reproduction. Lower cost plate making processes have recently become available via the use of laser technology.

Both flexographic and gravure are reel-fed (web-fed) printing processes. Both can use solvent type inking systems which are suitable for non-absorbent materials such as films and foils. Drying processes usually employ heated drying tunnels where the solvent is removed (and reclaimed). Special UV inks and UV curing systems, IR drying systems, etc. are also available.

With the flexographic process, water-based inks can be used on paper (original aniline dye process) where adsorption forms part of the drying process.

The choice of printing process depends on the design, the number of colours, the quality, the printing surface, the length of run, etc.

Heat sealing—peelable, semi-permanent, permanent

Basically any heat sealing operation depends on the correct combination of temperature, dwell time, pressure and removal of heat, with pressure being the least critical (note that if pressure is excessive, sealant can be squeezed out of the seal zone).

However, for an effective seal consideration must also be given to the following.

- Sealing jaw pattern (line, cross-hatch, etc.) may involve two matching patterns or one side smooth.
- Area or width of seal zone (if too narrow, the seal may not be totally effective).
- Condition of machine, evenness of seal pattern, alignment of jaws.
- Type of temperature control and operational range (thermostat control is better than simmerstats, ±7.5°C possibly drifting to 15°C ±with time). Electronic control is most accurate, say ±2.5°C.
- Product contamination—can interfere with sealing.
- Material to specification, e.g. no excessive caliper variation.
- How seal is achieved—platen or tangential contact (i.e. roller process where only a point of contact is made as distinct from a platen process where an area is sealed by a flat contact).

- If made from a reel process (which is usual), how resulting pack is removed from web by guillotining or punching. Hence accuracy and tolerance of cut are important so that an adequate heat seal margin is maintained.
- Number of joins in reel (should be flagged).

As a general rule 5 mm margins are recommended, but with push-through blister packs 3–4 mm is fairly normal.

Failures in heat seal can occur because of creases in the seal area giving rise to capillary leakage. Such capillaries are more likely to cause microbiological contamination than product losses. Under fluctuating temperatures the pack may breathe.

Seal defects may be detected by vacuum, pressure, pack deflection or gas sniffing.

In the case of multi-ply packs some plies (particularly the foil) can be perforated but, as other plies may be continuous, leakage will not be detected by a vacuum test. However, this perforation may be sufficient to affect product life. This type of leakage may have to be detected by visual means (microscope) or by careful separation of the plies by suitable solvents. The other alternative is to subject the pack to a cycling climatic test, i.e. 15°C 50% RH and 37°C 90% RH with 12 h cycles.

In some instances the heat seal ply of a lamination has to be peelable. These should be checked for peel strength over the shelf life of the product as in some instances the peel strength may reduce and so jeopardise the seal or increase it to a point where a permanent non peelable seal is obtained. The use of pattern lacquers also assists peelability.

Sealing can also be achieved by impulse, ultrasonic and RF/HF (radio or high frequency) methods and by cold sealing (pressure sensitive) type materials. See Chapter 11 for fuller details.

Lamination selection

Any laminate may consist of a number of plies selected from paper, cellulose, films, foil, coatings, tie layers, metallisation, etc. From previous pages the permutation possibilities are enormous. However this choice is restricted by:

- quantity/commercial availability
- technical requirements
- cost of base materials
- cost of lamination processes
- cost of printing cylinder and process (origination)
- the amount of laminate required (quantity)
- the yield (and from which choice the cost per area of laminate is derived).

One of the more complex laminates used consisted of clear LDPE/LDPE white/paper/LDPE/LDPE copolymer/30–50 μm, EVOH or Al foil/LDPE copolymer/ LDPE clear. As another example, many laminated tubes now consist of five layers e.g. two plies LDPE/40 μm foil/two plies LDPE.

By listing some of the more widely used flexible (lamination) materials it will be seen that certain materials are rarely used in laminations except for very specific and very specialised cases. For the purpose of this list, 'paper' will cover any variety.

- paper/PVdC and PVdC/paper/PVdC
- paper/LDPE and paper/Surlyn
- regenerated cellulose/LDPE
- foil/LDPE and foil/Surlyn
- foil/heat seal lacquer
- paper/foil/LDPE and paper/foil/Surlyn
- polyester/foil/polyethylene or Surlyn
- Nylon/polypropylene.

Note: Reference to specific gauges has been deliberately avoided, as final choice may depend on the many factors mentioned earlier. Surlyn is slightly more expensive than LDPE but gives a better seal if seal area is likely to be contaminated with the product. It also seals at a lower temperature, allowing increased output speeds.

Examples of pharmaceutical applications

Strip packaging

1 82 g/m^2 (25 μm) Al foil/30 g/m^2 LDPE or 25 g/m^2 LDPE. Gives excellent moisture, gas and light protection. External foil image.
2 30 g/m^2 glassine/ink/poly/67 g/m^2 Al foil (20 μm) 25 g/m^2 LDPE. Good protection— more subdued metallic image.

Sachet packaging

1 59 g/m^2 paper/14 g/m^2 PVdC—here PVdC provides protection and heat seal.
2 37 g/m^2 paper/glue*/24 g/m^2 Al foil (8 μm)/25 g/m^2 LDPE. Provides a good barrier and LDPE as heat seal medium.
3 24 g/m^2 foil/glue*/44 g/m^2 paper/37 g/m^2 LDPE. Similar to (2) but with foil external. See Appendix 9.1 for a typical QC specification for a Laminate material.

Blister packaging

Tray:

1 UPVC, 100–300 μm
2 PVdC coated (30–100 g/m^2), PVC or PET or PP (150–300 micron).

Lidding:

1 hard foil (18–20 μm), heat seal lacquer (3–15 g/m^2)
2 soft foil (25 μm), heat seal lacquer (6–12 g/m^2).

Food and confectionery packaging

The latest feature in food packaging is the retortable pouch as a replacement for an open topped can. At the moment economics are improving as the cost of steel and tinplate rises. However, filling speeds for pouches are considerably lower and additional strength outers (for stacking) add to the cost.

Retortable pouches can be made from combinations of foil, polyester, nylon, polypropylene, HD polythene, provided the laminant (or the process of lamination) employed will withstand the autoclaving conditions of temperature and moisture. Due to the flat shape of the pouch and high surface area, sterilisation times can usually be reduced compared with cans.

An important factor in both pharmaceutical and food packaging is that materials employed have FDA clearance with reference to any hazards. This may be checked by reference to the supplier and by obtaining full details of the formulation of each film employed.

The above are included as the pharmaceutical industry often follows the food industry when new materials are involved. This is sometimes economically sound where quantities are not sufficiently high to support the development of a specific material for the pharmaceutical industry, e.g. a retortable material in the food industry could be applied to a non-retortable use.

Other points

Laminates can be fabricated in a vast number of combinations and therefore the choice of the correct technical material may appear to be extremely complex. However, if the choice is restricted to what is adequate rather than perfect for a given purpose, it is possible to introduce a reasonable degree of standardisation and therefore obtain further economic advantages from larger demands.

An additional factor must also be considered. How do laminates compare with other competitive materials (such as glass, metal and plastic containers) in terms of disposal, pollution, reuse and conservation of the earth's natural resources? There is no doubt that combinations of plastic, cellulose, foil, etc. do present a disposal problem and although two of these can be incinerated, the economics of recovering the foil is relatively poor. The overall economics of using laminates can be compared

*12 g/m^2 LDPE is an alternative, extrusion laminated.

with other materials by quantifying the packaging costs and disposal/reuse costs. This approach does not include any 'convenience' value associated with each type of pack.

However, since most are reel-fed materials involving form fill seal activities, the space required for the storage of incoming materials is significantly less than for preformed containers which store air. There is also a significant saving in weight and space in many circumstances.

Life cycle analysis is expected to help in the long term the environmental aspects associated with the future of packaging. Life cycle assessment can also be applied. However, each involves factors which are difficult to accurately quantify.

Virtually all forms of flexible packaging find some use in either pharmaceutical or medical products. Pack forms include:

- conventional strip and blister packs
- cold formed foil blisters
- specially selected strip and blister packs with additional child resistance
- sachets and pouches (single and compartmental)
- sterilisable sachet and pouch systems
- a wide variety of overwraps.

Although the majority of the above will use conventional types and combinations of material, some will use specific or specialised forms which are solely related to pharmaceutical or medical products. Strip and blisters tend to fall into this latter category and are covered in Chapter 13.

Products and instruments requiring sterility need special mention, as both the materials and the processes offer a number of combinations. In total these can cover all the previously mentioned methods for obtaining a sterile pack, i.e. terminal sterilisation by steam autoclaving, or gamma irradiation, or the aseptic approach using dry heat, moist heat, gamma irradiation, accelerated electron or gaseous treatment, plus newer on-line aseptic processes. The last of these, which are currently being developed as a total inline system for the food industry, are normally reel-fed. The material is sterilised by UV or chemically by hydrogen peroxide or any similar sterilising chemical process, provided it is non-toxic with a low level 'residue'. In the USA hydrogen peroxide has been approved for the sterilisation of food materials with a residue limit of 1 ppm. This or a similar system is likely to find pharmaceutical applications.

Although certain products can be terminally sterilised by moist heat, gamma irradiation or accelerated electrons, these tend to apply to relatively few pharmaceutical applications where flexibles are involved (the last two are more widely used for medical instruments in pouches or sachets).

Moist heat is only a practical process where the product contains sufficient moisture and the seals are robust enough to withstand the temperature and pressure of the process. Stresses on the pack can be reduced by autoclaving under water and/or by using a balanced/overpressure autoclave. The latter is normally programmed to provide an external pressure during the cooling cycle in order to balance the internal pressure within the pack and thereby prevent the seals of the pack from rupturing. Paper plies (unless treated to repel water/steam) are usually excluded from pharmaceutical packs which are sterilised by steam. However, special paper plies with low porosity to prevent penetration by bacteria are available which are steam sterilisable, provided some degree of 'wrinkling' is acceptable.

Plastic 'papers' such as Tyvek, a porous spun bonded HDPE, are particularly suitable as an alternative to a sterilisable paper. Where non-porous plastics are involved sterilisation can be achieved by gamma irradiation, accelerated electrons, or gaseous treatment. Gamma irradiation can cause critical changes to some plastics, e.g. a less flexible material, discoloration or the release of gaseous substances. The last of these may give rise to organoleptic changes or increased risks with toxicity or irritancy, with a possibly greater emphasis on irritancy or allergenic type substances. Gamma irradiation can also change the structure of adhesives and alter the bond strength. It has been used to improve the peelability of some semi-permanent bonds.

Although ethylene oxide readily penetrates most plastics, attention has subsequently to be paid to any degassing phase and the level of residues associated with ethylene oxide, ethylene glycol and epichlorhydrin.

To conclude, it is likely these pharmaceutical applications for new combinations of flexible materials will follow those of the food industry, although occasionally there will be concepts which are specifically developed for pharmaceutical products. The purchasing power of the industry (compared with foods) will frequently restrict progress.

Increased growth in the materials currently used (listed below) is therefore more likely than a surge in the use of raw materials.

1 Foil:

- soft aluminium foil, 0.006–0.038 mm (as a ply in combination with other material)
- hard aluminium foil, 0.015–0.025 mm (as a ply in combination with other material)
- special foils for cold forming, 0.040–0.060 mm.

2 Paper: tissues, Kraft, bleached Kraft, glassine, glazed imitation parchment (GIP).
3 Plastic plies:

- LDPE, MDPE, HDPE, LLDPE (and combinations of these)
- Surlyn ionomer
- nylon and oriented nylon
- polypropylene (PP and OPP)
- Aclar
- Saran (PVdC)
- ethylene vinyl acetate
- polyester
- cellulose acetate
- regenerated cellulose
- polycarbonate
- ethylene vinyl alcohol
- polyvinyl chloride.

4 Coatings:

- metallisation
- polyvinylidene chloride
- nitrocellulose
- various heat seal coatings, washes, lacquers and varnishes
- solvent coatings
- silicon oxide and carbon coatings.

Conclusions

It is clear from the preceding text that polymers and coatings can provide a wide range of properties. Since most heat sealed (or cold sealed) flexibles are tamper-evident, this is obviously a positive feature which can encourage further unit dose type packaging. UK investigations have also established that, provided the materials are opaque or dark tinted and reasonably substantial (i.e. not flimsy), most strip and blister type packs offer reasonable child-resistance. This is generally as good as reclosable child-resistant packs where there is always a risk that the closure is not properly reapplied and any removal usually exposes a child to a greater quantity of a product.

It can therefore be concluded that there are many good reasons why the use of flexibles for pharmaceutical products should enjoy continuing success.

Other alternative coating processes

These alternative processes include metallising with metals other than aluminium, e.g. Camvac offers a coating of aluminium oxide on a material called Camclear. More recent additions include silicon oxide, (SiO_x) coatings, carbon like coatings, etc. Such coatings and volatile polymer coatings can be applied by a range of relatively new techniques found under the following headings: vapour, vacuum or electron beam deposition, sputtering and plasma enhancement, etc. (Figure 9.4). Coatings may involve gases, fine particles or a liquid which is usually deposited on the exposed material to be coated when it is passed over a chilled cylinder in a vacuum chamber. The thickness or weight of material deposited varies from very thin (measured in Angstrom) to that measured in microns.

Metallisation

This consists of fine particles of aluminium or other metals. Gain in barrier properties is often 20×or 40×but this reduces to 4×/5× if the material subsequently becomes severely creased and the surface is broken. Virtually any material can be metallised, e.g. PET, LDPE, PP, paper. They have a highly reflective surface but can be seen through against a bright light. Metallised materials can be laminated by wet or dry laminating systems, directly coated, etc.

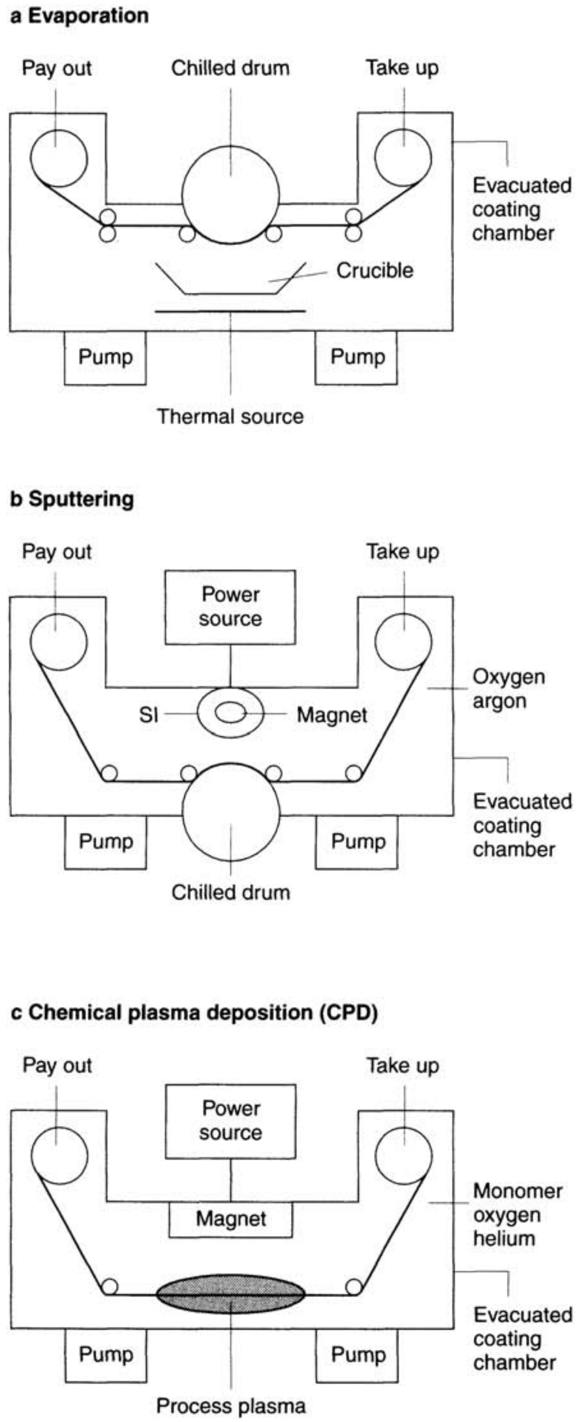


Figure 9.4 Alternative coating processes. In (A), the crucible contains material to be evaporated (aluminium, SiO/SiO_2, etc). Evaporation is by resistive heater or electron beam gun

Silicon oxide (SiO_x) coated materials (thickness 1500–3000 Å)

These are produced by several conversion processes (evaporation, sputtering, chemical plasma deposition). Evaporation is the same method as that used to create metallisation using aluminium. A material is heated in a crucible by either a resistive heat or an electron beam gun (hence the name electron beam deposition), whereby the material evaporates and subsequently condenses on a chilled film in a vacuum chamber. In the case of SiO_x coatings, the aluminium used in metallisation is replaced by SiO/SiO_2.

Sputtering (coating thickness 400–500 Å)

This process relies on an electromagnetic powered source which, together with an ionised gas mixture of argon and oxygen, causes the argon to bombard a silicon source whereby silicon atoms are created and subsequently attach themselves to a chilled substrate. The level of SiO_x created is controlled by adjusting the oxygen content. This process is the least economical (high energy required).

Chemical plasma deposition (CPD) (coating thickness 300 Å)

Although this can use a chilled drum, it is not essential, as the process involves a relatively low heat loading. The process again uses a vacuum chamber provided with a helium—oxygen mixture and a silicon-based monomer such as tetramethyldisiloxane or hexamethyldisiloxane. An applied power creates a plasma which activates the oxidation of the silicon gas, creating reactive chemical molecules which form the SiO_x coating on the film surface.

In general the CPD method, as used by Airco Coating Technology, gives the lowest deposit, a clearer film (others may have a yellow tinge), a better bond (chemical rather than mechanical), a more extensible material (stretches further before the surface ruptures), and this is achieved at a relatively low power input and a reasonable output speed.

SiO_x can be applied to films such as PET, PP, nylon and PE. O_x usually lies between 1 and 2 (but excludes 2, i.e. SiO_2). Improvements in barrier properties are up to 120× for oxygen and 45×for moisture, but severe creasing can cause a reduction. It has been shown that CPD coated SiO_x. films will withstand autoclaving (moist heat) of 121°C, or perhaps above.

Diamond-like carbon (DLC) (coating thickness in micrometres)

This is the newest coating process to emerge and has been applied to PET and PP films. The coating shows high hardness, transparency and impermeability, reducing moisture permeation by 40–60× and oxygen by up to 100×. In the process of manufacture acetylene is ionised in a vacuum chamber, releasing carbon onto a film.

Fluorination

Fluoride treatment of HDPE conveys improved resistance to solvent-type organics. Use for certain pharmaceutical products is under test, with manufacture of single and multilayer material including coatings.

Appendix 9.1:
Packing material specification

Notes

1. Where PHARMACEUTICAL COMPANY X HAS APPROVED BUYING SAMPLES FOR GOODS, any orders for goods of the same description subsequently placed by the Company shall unless otherwise indicated, be deemed to be placed on the basis of such buying samples as well as on the description and/or specification, and in all such cases the goods shall conform both to such buying samples and the description and/or specification.
2. The supplier shall effect no physical or chemical change to the product or its method of manufacture without the prior agreements in writing of the Company.
3. The Company reserves the right to revise or amend the specification after formal notification to the supplier.
4. The right is reserved to reject individual reels at the time of their use, should a fault appear within the confines of the reel.
5. Sampling, inspection and classification of defectives is conducted strictly in accordance to .... (The AQL’s for this are shown overleaf).
6. Visual defect classifications

CRITICAL

a) Admixtures
b) Print defects which result in non compliance with Statutory Regulations

MAJOR

a) Print defects which result in the illegibility of the text
b) Defects which are likely to materially affect the usability of the laminate when used on automatic packaging machinery
c) Defects which are likely to result in the product not meeting the required standard

MINOR

a) Print defects which do not affect legibility of the text but are a departure from normal commercial standards
b) Colour variation outside of the established tolerances
c) Defects which are not likely to materially affect the usability but are a departure from normal commercial standards

AUTHORISATION:
SUPPLIER: **DATE:**
RECEIVING COMPANY: **DATE:**
SUPPLIER