

Figure 2-3 - Densitometer

As in a spectrophotometer, light is reflected from the substrate under test, and the reflected light is passed through a lens or prism, and collected on a CCD. The CCD applies four filters, to calculate the reflectance in the cyan, magenta, yellow and 'black' regions of the spectrum. These filters will be applied electronically, and densitometers are also available to measure hexachrome and similar colour printing systems.

The reflected light is filtered to only analyse light over a certain wavelength band. Two examples are shown below, for cyan and yellow, Figure 2-4.



Figure 2-4 – Colour filters

The curves shown in each case represent the amount of light reflected from a sample at each of the wavelengths indicated. The box indicates the area over which reflectance is measured to calculate the density of the colour.

G.,

FAST FELT 2010, pg. 31 Owens Corning v. Fast Felt IPR2015-00650 The amount of light from the illumination source is known (incident light). The amount of light reflected is also known. Reflectance is thus defined as;

0

0

0

Reflectance = Reflected light Incident light

Equation 2.1 - Reflectance

Density is defined as the reciprocal of the reflectance, so that as the ink film thickness increases, and the reflectance decreases, the density increases. A logarithmic scale is used to improve the closeness of the results between density and ink film thickness. Thus density is defined as;

Density = Log_{10} (1/Reflectance)

Equation 2.2 – Density

This work was extended by Murray [19], who defined a technique for the calculation of the size of dots. By comparing both the solid density (D_s) (the density measured at 100% coverage) and the halftone density (D_t) (the density measured at the coverage under investigation) a relationship between the optical density and the actual printed coverage was observed to be;

$$Area = \frac{1 - 10^{-D_r}}{1 - 10^{-D_s}}$$

Equation 2.3 – Murray Davies equation

Close correlation was observed between densities measured with a densitometer and the fractional dot area (measured using an optical microscope). Comparing prints and proofs showed higher densities on the prints, caused by spreading of the ink into or over the substrate – tone gain. It was therefore demonstrated that tone gain may be

calculated from the optical densities.² Yule and Nielson [20] modified the Murray-Davies equation to include an empirically derived factor to compensate for light scattering within the substrate. This attempts to calculate a value for the physical tone gain observed in the sample, ie the degree of spreading observed by the ink on the substrate, making dots larger than was intended. This method requires a visual assessment of a 50% coverage dot, and as such requires a significant degree of user intervention and thus an additional level of variability. For the purposes of this work it is attempted to minimise user intervention, and to use properly defined techniques. As a result, this technique was not used for the calculation of physical dot gain. Image processing provides techniques for the calculation of physical dot gain over the range of tonal coverage [21].

However, conventional colour measurement techniques were used, in evaluation of scumming, and these are discussed fully in Hunt, [15]. Using optical spectrodensitometry to measure colour, both in terms of the printed ink, and in the unprinted areas enabled identification of the levels of scumming between the printed substrate (in unprinted regions) and the bare, unprinted substrate.

2.2.2 Surface Profiling and Volume measurement

0

0

A gravure cylinder is an engraved, etched or ablated surface, and one focus of this work was to analyse and measure the engraving details on the cylinder. As such, this section will review the methods available for measuring surface topography, with particular reference to those methods that are most appropriate. [22]

Many techniques are available for surface profiling, from the most complex (AFM, STM etc) to simply dragging a fingernail over a surface. Each method has its own advantages and disadvantages, but since the purpose of the use of surface profiling in this case is to investigate indentations, intentionally placed into a surface, rather than natural indentations and ridges, the deflection that must be measured may be large and

 $^{^2}$ Tone gain is defined as the calculated tonal coverage minus the specified coverage. Thus a 50% dot, measured to be of 58% coverage exhibits 8% tone gain.

the sides may also be relatively steep. As a result, many of the methods traditionally used in engineering for surface profiling will not be applicable.

0

Traditionally, stylus measurement techniques are used in the analysis of surface profiles [23]. In these procedures a stylus is dragged across the surface being analysed whilst in contact with the measurement surface. The vertical displacement of the stylus is tracked, and a profile is generated. From this data, various roughness parameters may be calculated, eg. R_a , R_q , R_z , R_t etc. [24]. This two-dimensional data, although useful for calculating the roughness of a surface, is of little use when attempting to calculate volumes, which require three-dimensional measurements, although a raster scan may be performed to produce a three-dimensional measurement. However, this is not suitable when measuring steep cell sides as under these conditions the stylus sides contact the indentation, and not the tip of the stylus.

Several different three-dimensional methods have been proposed for analysis of gravure cells, many of which due to the nature of the cells are impossible to use. Kunz [25] simply cut cylinders up to examine them under an optical microscope. Due to the cost involved in this sort of destructive analysis, little other work has been published using this method, although it does allow reasonably detailed analysis. Scanning electron microscopy (SEM) was also abandoned due to the necessary destruction of the cylinder to obtain suitable sample sizes.

Certain stylus techniques are applicable to three-dimensional measurements. Bowen et al [26] [27] discuss the uses and limitations of atomic force microscopy. Atomic force microscopy (AFM) uses a very fine stylus mounted on the end of a cantilever, held close enough to the measurement surface that inter-atomic forces cause the stylus to be deflected. This deflection is measured using a laser, reflected off the gold plated top surface of the cantilever, and received by a position sensitive detector. This deflection is interpreted into a height profile of the measurement area. It is possible to operate an AFM in several modes, including contact (where the stylus is held in contact with the surface), tapping (where the stylus is briefly brought into contact at each measurement point, but is not dragged between them) or non-contact (where the atomic forces are measured to keep the stylus and the substrate separate). The stylus is moved across the surface of the substrate in a series of linear measurements, which are all taken to produce a three-dimensional image. It is however, only applicable to maximum height deviations of $\pm 10\mu$ m, due to the length of the stylus protrusion from the cantilever and thus not usable to examine cells, which may be up to 100μ m deep. Scanning tunnelling microscopy (STM) is another probe / stylus type measurement, where the probe is maintained close to the substrate, with either a constant 'tunnelling current' being passed between the stylus and the substrate (which must be electrically conductive) or with the stylus height being maintained, and the current being measured to infer the distance between the stylus and the substrate. This was excluded for similar reasons [28], and particularly time – it will require approximately 1½ years to measure the area of a single large cell, although this will allow calculation of the position of each atom in the outer surface of the cylinder chrome surface.

White light interferometry [24] is a recent development, and allows a non-contact examination to be performed. The principles of interferometry are well established, but the use of a white light source giving a much longer coherence length and the use of an LVDT controlled head movement allow much larger scan ranges, and with minor modifications to the optical systems (detailed in chapter 3.2) allow detailed analysis of the top surface and insides of gravure cells, including measurement of the depth, volume, open area, width, and length of the cells.

In white light interferometry a beam of white light is split by a beamsplitter, with half being passed to a reference mirror, and half being passed to the substrate. These beams are then recombined, and the resultant interference pattern is captured by a CCD camera, Figure 2-5

20



Figure 2-5 - Interferometer

Light comes from the illumination source, (A) (normally a standard tungsten-halogen bulb) and is divided into two beams by the beamsplitter, (B). Half the light is sent to the reference mirror, and half to the substrate. The reflected light from the mirror and the substrate is then recombined, and passed to the CCD array, (C). The whole apparatus is then slowly moved closer to the substrate, and images are regularly captured for analysis by computer.

10

0

It is possible to operate most interferometers in two modes, VSI (Vertical Scanning Interferometry) and PSI (Phase Shifting Interferometry). PSI is the older technique, having been used in interferometers since the early 1980s. It uses a series of measurements, where the apparatus is moved only ¹/₄ of the wavelength of the light being used between each measurement. From this measurement, the relative height of each pixel is calculated. Due to this limited number of measurements, the speed of measurement is significantly higher than when taking VSI measurements. The limitation of these measurements is in terms of the maximum height differences which can be measured, 160nm in PSI mode, 500µm in VSI mode. Vertical resolution is improved in PSI mode, typically 3Å while with VSI measurements a resolution of lnm can be achieved with care. The latter is very appropriate in the current application and so VSI measurements were used throughout this investigation.

FAST FELT 2010, pg. 36 Owens Corning v. Fast Felt IPR2015-00650

2.2.3 Ink transfer measurement

0

0

0

0

Since colour is a function of ink deposit, which is itself a function of print quality it is necessary to analyse the quantity of ink being transferred. Deposit is determined by transfer mechanisms and motivates this review.

Kistler and Schweizer [29] describe several techniques for film thickness / ink transfer measurement. Absorption techniques, either using infrared or microwaves may be used to examine the thickness of transferred films, whereby the absorption of either infrared light or microwaves may be measured, and a thickness may be inferred from the consequent attenuation. This technique however, requires greater film thicknesses than those observed in the gravure process due to the accuracy of the system, and the need for enough of the film to be present to absorb the infrared/microwaves. X-ray fluorescence measures the emission of X-rays from suitable elements. Disadvantages of this method include extremely long sampling times, which are required to acquire accurate mean values, and the necessary use of unrealistic inks containing tracer elements for testing purposes.

Capacitance gauging is also occasionally used for analysis of thin films. By placing a flat plate sensor above a grounded target plate, with the substrate between them, the capacitance of the parallel plates can be measured, and the thickness inferred from the change in capacitance between the unprinted substrate and a printed substrate with a known film thickness applied. It is however, impractical to use this technique with films of the thickness used in the gravure process due to the low precision of the technique. Each of these three techniques is only able to be used with even films, such as are obtained at 100% coverage. They are also only able to be used over relatively large areas, of the order of a few mm² at least. Due to the need to measure much smaller areas for the analysis of individual dots, these methods were not usable.

The Moiré technique (also sometimes referred to as 'mechanical interferometry') uses interference techniques to measure film thickness, however it is limited to use on films of greater thickness than $10\mu m$, and only has a vertical resolution of $10\mu m$. By superimposing two optical gratings, one undistorted, and one distorted by the film, it is possible to infer the thickness of the film. Again, this is only of use with significantly larger areas than are available and significantly thicker film deposits.

0

0

0

'Profilometers are typically not used for film thickness measurements' ([29] p226, 7.3.2.1) because traditional profilometers use a stylus that is dragged across the surface of a sample. Optical profilometers however, are extremely suited to the measurement of films. Two primary techniques are available. The triangulation method uses a laser beam, focused on the surface at test and viewed at a fixed angle by a position detector, similar to the technique used for height displacement measurement as used in an atomic force microscope. As the height of the surface changes (i.e. as the thickness of the transferred film changes) the light spot on the position detector moves, thus giving a measurement of displacement of the test surface, relative to the detector. The method can use extremely high sampling rates (up to 40kHz), high resolution, large measurement ranges (up to 10mm) and a relatively high working distance (up to 5cm). Unfortunately it is only practical to use this on thicker films than are transferred in the gravure process. White light interferometry [24] however provides a non-contact, non-destructive technique for the analysis of films. The technique is described in 2.2.2. By varying the optical configuration of the interferometer, suitable x-y measurement areas may be defined either for the measurement of individual dots (the gravure process not being a contone process) or for continuous films, whilst the vertical resolution of the interferometer allows for the measurement of the extremely thin films observed. This technique does have several drawbacks, primarily those of time and cost. Measurement of film thickness takes as little as 30s, but measurement of film thickness where a broken film occurs (for example, at the highlight end of the tonal range) takes approximately five minutes per sample. This is, however the only process which allows the measurement of such broken films [30].

When calculating the quantity of ink released from the cells it is necessary to either know the amount of wet ink that is transferred, or to calculate the volume of dry ink, and the amount of solvent which is associated with this quantity of solids. Due to the extremely fast drying nature of the ink, these wet measurements are not possible, and as such dry analysis is required. A precise breakdown of the ink was supplied by the

manufacturer, and the quantity of solvent in the wet ink is known precisely, but the solvent content in the dry ink must still be measured. Traditionally, to identify the solvent component of a sample, gas-chromatography mass-spectrometry is used [31] [32] [33]. Thermal desorption heats the sample, driving off the volatile fraction into a moving, inert gas stream, and condenses it onto a cold surface [34] [35]. This surface is then rapidly heated; releasing the volatiles back into the gas stream, and passed onto the gas chromatograph - mass spectrometer. Clearly the thermal desorber is required to ensure that all the volatile components are injected into the gas chromatograph at the same time, rather than over a period of several seconds if the entire sample had to be heated. The sample is then passed through a column, which separates out the components. This is then passed on to the mass spectrometer, to identify the components that are included [31] [36]. The mass spectrometer uses ionisation techniques to break up the component molecules, and then analyses the components by comparing the break-up pattern with those of known substances. This is performed automatically. Once the quantity of solvents in the sample has been measured and the volume of the dry dots has been calculated, it is possible to calculated the quantity of solvent originally related to the solids, and thus the volume of wet ink which was released from the cell.

0

0

٢

Gas chromatography has been used as an analytical tool since approximately 1950, although experimental tools have been used since 1940. The sample under test is injected into a moving stream of gas, which passes through an oven. Inside the oven is a long coiled tube, which the gas passes through. By the end of the tube the components have been separated by elution. They are distinguished by the different times they take to pass through the column – the retention time.

Finally, the sample is passed to the mass spectrometer. This is the oldest part of the system, with mass spectrometry having been used in some form since 1898, and in conjunction with gas chromatographs since 1958, shortly after its introduction. A mass spectrometer takes the input molecules and bombards them with electrons to make ions, which spontaneously break up. This break-up pattern is specific to each molecule, and so a piece of software may analyse the patterns observed, and identify the most likely chemicals from the pattern. In addition, the frequency of each ion (or abundance) is measured, thus allowing a detailed breakdown of the components fed to

FAST FELT 2010, pg. 39 Owens Corning v. Fast Felt IPR2015-00650 the mass spectrometer. Results are produced as a 'total ion chromatograph' (TIC), which details the frequency of each ion, and each fragment identified from the breakup of the molecules. Automated analysis follows, identifying each component in the sample, and its abundance from the TIC.

There is no published literature indicating that this technique may be used on printed polymer films for this specific purpose. Similar trials have however, been performed, for example Garcia and Sanz [37] who used the procedure to analyse the volatile content of *Origanum Vulgaris* leaves. For the purposes of this research, a new technique was developed in conjunction with the manufacturers of the thermal desorber – gas chromatograph – mass spectrometer in use. [38]

2.3 Experimental Results

To consider the mechanisms of ink release from gravure cylinders, the review of previous experimental work relating to rotogravure printing is discussed in three sections. The first details the effects of altering inks, the second of changing substrates, and the third examines the effects of cylinder engraving.

2.3.1 Inks

0

0

Rotogravure inks are predominately solvent based. They consist of a series of components intended to adhere to the substrate, and are approximately 80% solvent by mass. Much of the rest of the ink is made up of pigment and binder. Many things may be added to the basic solvent blend, including adhesion promoter (itself another solvent), nitrocellulose or polyvinyl butyral, surfactants and many other chemicals specific to an individual ink. Changing any one of these components may significantly affect the ink, and thus its transfer.

Bohan et al [39] investigated the effects of changing ink viscosity, demonstrating that ink viscosity has a large influence on the colour of the final printed copy with ΔE values of up to six being recorded. These changes are significantly larger than those

FAST FELT 2010, pg. 40 Owens Corning v. Fast Felt IPR2015-00650 necessary to be detected visually, and point to the importance of ink viscosity It was found that subtle changes in viscosity at low viscosities are far more significant than changes at higher viscosities. These experiments were also repeated, with the same findings in Bohan et al [40]. Kunz [25] examined the effects of viscosity on print, and observes that tone gain is significantly increased at low viscosities, while it is reduced at high viscosities. He also notes the effect of changing viscosity on the filling state of the cells, where high viscosity inks do not fill gravure cells as completely as low viscosity inks. The analysis technique used for this is not entirely clear, but it is known that he used an early interference technique to calculate the deflection between the top surface of the ink and the top surface of the cylinder.

Further mention is made of ink temperature, and its effect on ink transfer, although this is primarily due to temperature affecting the viscosity of the inks under study. It should be noted that the inks Kunz used are of a significantly higher viscosity than those which would be used on a press today (36s, Zahn 2 – normal operating viscosities are of the order of 20s, Zahn 2).

Elsayad et al [41] investigated the effects of viscosity and resin content, discovering that the amount of ink transferred by the cylinder increased as the viscosity of the ink increased. It was also found that the type of ink resin used had a considerable effect on the amount of ink transferred. Benkreira et al [29] [42] [43] [44] also investigated the effects of ink, indicating that it has little effect on the release.

Pekarovicova et al [45] investigated the use of hot melt inks for gravure printing, but as their research was limited to inks which at best were several hundred times more viscous than gravure inks, not solvent based, and limited to draw-down testing of them, this has little relevance to the gravure process.

2.3.2 Substrates

0

Gravure printing may be performed on many different substrates, from extremely thin papers, such as those used in catalogue printing, to heavy papers as used in art books. From thin packaging films (which may themselves be metallised or coated in some other fashion) to thick cartonboard as used in cigarette packs. All the work in this investigation was performed on plastic substrates, due to the non-porous nature of plastic films, but even here, by changing the type of film, the surface roughness and specific surface energy may change significantly. The specific surface energy relative to the surface tension of the ink may well need to be changed, using (for example) corona treatment, to enable the ink to transfer and adhere.

0

0

Kunz [25] indicates that the substrate is an extremely significant variable. He indicates that there is a "*clear connection between smoothness and printability*", suggesting that there is an operability window for smoothness, where substrates that are either too rough, or too smooth will have "*negative effects*"³. It is also observed that for printing to be achieved, it is necessary for the impression roller to force the substrate into contact with the ink, and that this is "*problematic*" due to the need to squeeze the substrate into the cells, as the meniscus of the ink lowers the top surface of the ink below the top surface of the cylinder. However, this work was performed using ink significantly more viscous than that used in the modern process (34s Zahn 2) and at significantly lower speeds than those used on commercial presses.

LaFaye et al [46] produced different papers and printed them on test equipment (some were also printed on commercial equipment) to test their printability. They show that both solids content and coating weight (both of which promote surface smoothness) have a positive effect on printability. This correlates with [25], where it is shown that generally smoother substrates print better. Heintze [47] also investigated the smoothness of coated board on a GRI printability tester. He discovered that the wear of the doctor blade can affect the print quality, particularly in terms of the 'speckle' of the print. He acknowledged that the use of a lamella blade minimises this problem.

Hansson and Johansson [48] [49] examined the printability of different substrates using stereo photometric techniques, using irradiance to infer a measurement of surface height and roughness. They also determined that areas of substrate significantly lower than the average surface tend to have missing dots. This method is

³ The comparison is given to water on glass, which beads up and does not adhere

however, not as accurate as white light interferometry, which was used in the conduct of this investigation.

2.3.3 Cylinder analysis

0

0

Tucker [50] examined the use of interferometry to evaluate anilox rolls, but since an anilox roll is an intaglio roller, rotating in an ink bath, the principles of measurement hold for measurement of gravure cylinders. Interferometry was considered to be a breakthrough in measurement technology, replacing the traditional methods of theoretical calculation, and liquid volume measurement. Theoretical volume measurement is the standard technique where an optical microscope is used to calculate the open area at the roller surface, and to infer the volume from these physical dimensions. It is limited by the operator's identification of edges, and the assumptions that must be made about the shape of the cells, which, for the purposes of calculation must be assumed to be ideal, despite the limitations which this imposes. Liquid volume measurement is an indirect technique, by which a known volume of liquid is applied to the cylinder, doctored, and then 'printed' onto a substrate. The area printed can then be calculated, and the volume inferred from this measurement. This again, is limited by the operator as the doctoring process is likely to influence the area over which the liquid is spread and calculation of the printed area is dependent on the users interpretation [51]. It also assumes that the cells are completely filled, which has already been shown by Kunz [25] not to be true.

2.3.4 Ink Transfer

Several variables are believed to influence ink transfer. Of these, only engraving type and screen ruling have been investigated. This section reviews the work that has been performed in this area. This section also includes scumming as this can be considered as unwanted ink transfer.

Little work has been performed in evaluating the different forms of engraving in terms of the ink release. Kunz [25] however completed the earliest work, and he

examined the filled and emptied states of four different types of cells, establishing that laser engraved cells release the most ink (88%), followed by two types of etching, round dot (80%) and conventional (75%), with electromechanically engraved cells releasing the least ink (68%). This he attributed to the shape of laser engraved cells, which have very smooth bases and lack the pointed apex of electromechanically engraved cells. This was, again, established by the use of an early interferometric microscope, but under conditions far removed from the modern industrial process.

0

0

Pulkrabek and Munter [44] examined the release of ink from a knurled roller, analogous to a gravure cylinder. Their work indicates that 59% of the volume of the ink in the roller grooves is transferred to a substrate, but that the release is inversely proportional to the fluid viscosity to "*a small degree*". It should be noted however, that their ink was highly shear thinning, with a rest viscosity of 5000cP (approximately 20,000s – Zahn 2) – or approximately 1000x more viscous than normal process gravure ink, and a thinned viscosity of approximately 100cP (43s - Zahn 2) – or approximately twice as viscous as normal gravure ink. No indication is given of how the portion released was measured.

Benkreira and Patel [42] measured transfer using infrared absorption techniques in reverse gravure coating. It was discovered that the amount of ink released from a cell appears to be consistent at approximately 32% of the transferring cell volume. By using three different types of cell engraving, it was also established that the release is independent of the type of cell, except at very low speeds, where variation was observed. Benkreira et al. [43] measured the transfer from both loaded and unloaded forward gravure cylinders, establishing that unloaded cylinders release approximately 20% of their volume, but that loaded cylinders release up to 33% of their volume. It should be noted that no indication of the impression pressure was given, despite significant evidence that the impression pressure will affect the release from the cylinder. Benkreira and Cohu [52] continued this work, demonstrating that with unloaded forward gravure coating release is of the order of 15-20% of cell volume. This work also demonstrates that the thickness depends on operating parameters. including speed and cell geometries. It should be noted that all this work was carried out on test rigs, not on production scale presses. It should also be noted that the infrared analysis equipment used in all three of these studies provides an accuracy of ± 0.5 um – or approximately 10% of the thinner film thickness measured. The technique used for analysis relies on infrared absorption over a large area, and averaging over a period of time. As a result, the technique is not applicable to the measurements being performed here. Furthermore it should be noted that all this work has only been performed on solid coverages, and not the halftones being examined in this work.

0

0

Jeske [53] simulated the gravure process with an impulse driven press, where a loaded mass forced the substrate onto the printing plate. Although this is similar to the earliest gravure printing techniques, several problems occur with this technique. Firstly, the printing plate forces the substrate into a completely different contact regime than a cylinder. Secondly, the plate was doctored by hand, despite Kunz's [25] and others assertion that doctoring can, and will affect the filling state, and thus the release characteristics of the cells. Despite the criticism that "unrealistic inks" were employed in the research of other authors, Jeske used a highly viscous ink, mixed with oils to reduce the viscosity, rather than solvents. No indication is given of the size of the measurement area, but analysis of transfer was calculated by the differential mass method, the accuracy of which is low, particularly for practical conditions. It is also known that the surface tension of the ink affects release to some extent, as the ink is 'sucked' onto the substrate (Kunz [25]) yet the inks used here, have a significantly higher surface tension than inks made with solvents. It is also demonstrated here, through the use of photomicrographs that the cells are not completely filled, although the technique used to demonstrate the lack of filling does not allow quantification of the fill state.

Kapur, Gaskell and Bates [54] investigated the release from a gravure cylinder in an offset gravure coating test rig. Tests were performed under conditions similar to production conditions. This process is similar to the gravure printing process in that it uses forward transfer from the cylinder to an offset roller, albeit with reverse transfer to the web from the offset roller. Ink was collected from the offset roller over a known time period to discover how much ink was being transferred, a far more accurate technique than the absorption techniques employed by other researchers. It was shown that release from the electromechanically engraved roller was approximately 15% while higher release was obtained from a coarse screened laser engraved roller (20%).

Lower values were obtained for higher screened laser engraved rollers. It should be noted that this experiment is the one closest to both commercial production conditions, and to the conditions used in this investigation.

However, in each case considered above, theoretical calculations were used to establish the volume of the cells. If this is not the case, then significant errors in the release calculation have been included. This uncertainty motivated the measurement of actual cell volume as described in this work.

2.3.4.1 Scumming

0

0

0

As explained, scumming is ink transfer from non-image areas and is a key process problem. Since little published work exists, the most relevant is blade coating, which is used to transfer thin continuous films.

In blade coating, a flexible blade, analogous to the doctor blade used in the rotogravure process is used to meter the fluid being coated onto the substrate, and the principles are discussed in [29].



Figure 2-6 - Blade coating response (From Kistler and Schweizer [29])

FAST FELT 2010, pg. 46 Owens Corning v. Fast Felt IPR2015-00650 As the blade is brought closer to the substrate surface, the blade initially remains straight, reducing the coating thickness (moving from 'A' to 'B' in Figure 2-6). However, as the blade is brought closer, the hydrodynamic forces under the blade increase, reaching a maximum, and forcing the blade to bend (point 'B'). At this point the tip of the blade moves away from the surface, allowing a thicker coating to be laid down, despite the increase in blade load being applied. A maximum deflection causes a local maximum in film thickness (point 'C'). Further increase of the blade load brings the bent blade closer to the surface, reducing again the thickness of the applied coating (point 'D' onwards). It indicates that blade loading conditions applied by the printer may lead to unexpected results, as small adjustments to the blade load may increase the film thickness passing under the blade (moving from 'B' to 'C'), decrease the thickness (moving from 'A' to 'B') or make little difference, for example, the line on Figure 2-6, indicated 'E' crosses the curve at three points. Where the line crosses the curve, the same film thickness will be printed, despite the line crossing at three significantly different blade loading conditions, Kistler and Schweizer, [29].

2.4 Numerical Modelling Experiments

0

0

0

Although this investigation is experimental, numerical investigations are considered for completeness, since they often provide useful physical insight.

Only a few studies have been reported, however Benkreira and Patel [55] modelled ink release from several different roll configurations, including trihelical, quadrangular and pyramidal, noting that the ink release appears to be independent of the configuration of the cells and that one third of the ink in the cells will be transferred to the substrate. They also observe that at low Reynolds numbers (defined using substrate speed as the characteristic speed and cell volume divided by cell open area as the characteristic length), a higher release may be obtained. Schwartz [56] used paraboloidal cells, rather than the more usual trapezoidal/pyramidal cells. He observed that a number of parameters will affect the ink release, but does not quantify this remark. He observes that about 57.3% of the ink appears to be released from the cells. It is noted that this value is significantly higher than other calculations, although no explanation for this is offered. Both studies used computational fluid dynamics to examine the flow of fluid between cell and substrate, and Benkreira and Patel compare this with their own, experimentally established data.

Powell, Savage and Guthrie [57] used Langragian finite element analysis to examine the release of Newtonian fluids from trapezoidal cavities in two dimensions. Although a model is demonstrated, no relevance is drawn to the commercial process. It is demonstrated that according to the model, approximately 90% of the ink in the cell will be transferred, but that as speed is increased, the quantity of ink transferred will decrease and by inference that as viscosity decreases, so the amount of ink transferred will decrease

2.5 Closure

0

Several studies have been reviewed concerning ink release, and the technologies required to examine and measure it. A wide variety of results have been found, stating the portion of ink released to be anywhere between 15% and 90%, with several stating that ink release is of the order of 33%. Many of these trials however, have used unrealistic conditions, and all have required the measurement of continuous film transfer due to the measurement techniques employed. It is also clear that it has not been possible for accurate measurement of the cells due to a lack of the necessary equipment to do so. This inaccuracy may well lead to significant errors in the measurement of release.

Ink release from non-image areas has received little scrutiny, and as such blade coating has been reviewed. This is because scumming is attributed mainly to the factors that affect the doctoring process that bears a close linkage to the blade coating process..

Against this background, the work in this thesis will focus on establishing ink release from the gravure cylinder and will also focus on factors that influence this release. The process of transfer from non-image areas will also be explored.

Chapter 3 Methodology

3.1 Introduction

Ċ.

1

Despite its apparent simplicity, gravure printing is extremely complex, with many factors affecting the process. A brainstorming session with a group of gravure professionals indicated a list of well over 100 variables, and these are categorised in Figure 3-1, below.



Figure 3-1 - Variables in the Gravure process

Many of these variables will also have sub-variables (for example, engraving type could include screen ruling, stylus angle, compression ratio and others). An experimental programme that addresses all of these will be extremely difficult to plan, and so strategically this work focussed on the cylinder and its effect on image transfer, while maintaining the remainder relatively constant within practical limits. The parameters that were included in this study are shown in red text in the variable

FAST FELT 2010, pg. 49 Owens Corning v. Fast Felt IPR2015-00650 list in Figure 3-1. These have been chosen, since professional experience suggests that variables in the ink, substrate and process interact strongly with the cylinder.

Experiments were carried out to explore the mechanisms affecting image transfer, in the form of the ink release from the cylinder in the rotogravure process. This chapter focuses on the methodologies used within the investigation, both in terms of the experimental and analytical aspects. It is divided into three sections, relating to the measurement of printing cylinders, engraving diamonds and prints. The first section outlines the theory and techniques of the interferometric methods used in the measurements performed on printing cylinders. This also includes detailed information on the conditions used in the analysis of the cylinders. The second section discusses the interferometric measurement of engraving diamonds in terms of their shape and characteristics. The final section discusses the techniques used in analysing the prints from the investigation. Prints were analysed using interferometry (to calculate the volume of dry ink on the substrate), spectodensitometers (to measure the colour of the prints) and thermal desorption - gas chromatography - mass spectrometry (or TD-GC-MS) (to quantify the amount of solvent that remains in the 'dry' ink, and substrates, printed and unprinted). The use of interferometry and spectrophotometry are detailed in this chapter. The use of a TD-GC-MS is demanding, requiring extended effort and exploration to be able to develop methods for the testing of new substances. As a result, a chemist was involved with the analysis of the susbtrate, and as such only a brief synopsis of the method is given in Chapter 4.

Several challenges needed to be overcome, not least the requirement to be able to measure cells with precision and on site, requiring the development of a new technique to analyse the cells This was (according to the manufacturers) not possible with current technologies. In addition, it was necessary to develop a new technique to quantify the volume of ink transferred onto the substrate, and for new techniques to be developed to analyse the quantity of retained solvents in the dry inks – this necessitating a great deal of development to identify the solvents and their quantities in the prints / substrates / inks.

35

3.2 The Cylinders

0

Four cylinders were produced specifically for the experimental trials involved in this investigation. This section details the design of the images engraved onto them, the surface finishes applied and the analysis of the cylinders.

3.2.1 Cylinder Production

The first cylinder (as used for the investigation of ink release from image areas) was engraved on an Ohio Electronic Engravers engraving machine. The second, third and fourth cylinders were engraved on a Daetwyler Gravostar engraver. All cylinders were polished, pre-engraving on a Daetwyler Polishmaster, and finished on a Daetwyler Finishmaster. The cylinders were proofed to check the engraving before printing. The test image is shown in Figure 3-2.



Figure 3-2 – Test image

The cylinder comprises five primary areas, identified in Figure 3-2 as A to E, each designed to be a small enough section to be measured using an automatic spectrophotometer. Each section contains a series of gradation scales, engraved to cover the standard range of compression ratios, screen rulings and stylus angles.

Several other features and measurement aids were built into the image. These are identified and detailed below.

Each individual gradation scale required the engraving machine to be stopped, recalibrated and restarted. In addition, four different engraving diamonds were used (to investigate the effects of stylus angle). Total engraving time was approximately 30 hours.



Figure 3-3 - Test image with identifiers

- A. Blocks of gradation scales. Each represents a different set of engraving conditions. The coverage goes from 0% to 100% and back to 0% to allow analysis of the effects of wipe direction. Three repeat engravings are also included to investigate any differences across the width of the web.⁴
- B. The engraving conditions related to the gradation scale directly above the identifier.
- C. Level of coverage (10%, 20% etc)

Ó

- D. Cell to wall ratio analysis patches
- E. Gradation scales. Investigating the effects of changing coverage across the web rather than along it.
- F. Blank patches. Used for paper calibration of the spectrodensitometer.
- G. Alignment targets. Used for lining up the automatic spectrodensitometer.
- H. A wipe line. Used to identify between images, and included whenever possible in the gravure process to ensure a clean wipe from the doctor blade.

⁴ Full details of the engraving specifications are given in Appendix 2

3.2.2 Cylinder Design

0

3.2.2.1 Ink Release from Image Areas

Due to the significant expense involved in the production of a gravure cylinder, it was necessary to significantly over-design the cylinder, thus allowing further investigation to continue beyond that used in this investigation. In this particular work, three screen rulings, three compression ratios and three stylus angles are investigated, along with their effects. The test image however, was planned to incorporate significantly more data. As a result, it was necessary to incorporate different experimental design techniques.

Traditionally, full factorial experiments have been used to investigate the effect of changing variables, and the number of experiments required is equal to the number of levels of each variable multiplied together. Thus;

Three variables, each at three levels = $3 \times 3 \times 3 = 27$ experiments

Given the four stylus angles, six screen rulings and seven compression ratios engraved onto the cylinder, this would require $(4 \times 6 \times 7 =)$ 168 separate gradation scales, and would cost (conservatively) £15,000 to engrave. This would also require an estimated four additional years of measurement, and so the application of experimental design is justified.

There are three primary techniques used to reduce the number of experiments that need to be performed: experience, elimination, and orthogonal array techniques.

Experience relies on an understanding of the process. A set of variables is reviewed and reduced in number, until a full factorial experiment becomes practicable. Unfortunately, in processes such as gravure printing where the process is not well understood this can easily lead to the elimination of significant variables. Elimination requires a logical approach to the problem. Each variable is considered on its own, and independent of all other variables. The variables are prioritised, in terms of the size of their effects (as believed by the experimenter). The first variable is then explored, holding all other variables constant. Once this one has been done, the second may be explored and so on, until the process is explored fully. This does however require two things. Firstly, it assumes that there is no natural variability in the process, and that identical trials will lead to identical results. Secondly it requires the assumption that there is no interaction between variables [58].

As a result, the need to reduce the number of scales being used was examined via the use of orthogonal arrays. The use of orthogonal arrays has been used on many occasions to reduce the number of experiments used while fully investigating the effects of a large number of variables [59] [60] and has been applied to printing by many investigators. A typical example is Bohan et al. [38].

By using orthogonal array techniques, it is possible to investigate large numbers of variables in significantly less experiments. For example, using an L8 array, it is possible to investigate seven, two level variables in eight experiments. Traditionally, this would take 128 experiments. As a result it is possible to significantly reduce the time and expense involved in experiments.

As three levels of the variables were to be explored, L9 arrays were selected. Although these allow the analysis of four, three level variables, they only allow the use of two, if the interactions between them are to be assessed. However, by using multiple arrays, it is possible to 'nest' them, cutting down the number of experiments. An example of an L9 array is shown below, Figure 3-4

0

0

0

Experiment	Vari able			Result	
Number	1	2	3	4	1
1	1	1	1	1	####
2	1	2	2	2	####
3	1	3	3	3	####
4	2	1	2	3	####
5	2	2	3	1	####
6	2	3	1	2	####
7	3	1	3	2	###
8	3	2	1	3	####
9	3	3	2	1	###

0

0

0

Figure 3-4 - L9 array

For each experiment, the table is read from left to right. The first column merely indicates the number of the experiment, from (in this example) one to nine. The next four columns indicate the variables being used, and the levels to which they should be set for the experiment. The final column indicates a quantitative result. This may be printed density, cell volume or some other quantifiable variable. An array, indicating the use in practise is shown below, Figure 3-5

Experiment		Vari a	ble		Result
Number	CR(°)	SR(Ipcm)	3	4	1
1	37	60	1	1	A
2	37	70	2	2	В
3	37	80	3	3	С
4	45	60	2	3	D
5	45	70	3	1	E
6	45	80	1	2	F
7	57	60	3	2	G
8	57	70	1	3	Н
9	57	80	2	1	1

Figure 3-5 - L9 array. Compression ratio and Screen ruling

In this example, two variables have been assigned to the first two columns, CR (compression ratio) and SR (screen ruling), while columns three and four have been left blank. This was done in order to identify any interactions between the variables. None were found.

By averaging results A, B and C, a value can be assigned to (for example) cell volume at 37° compression ratio. By averaging D, E and F, and G, H and I, it is possible to

assign values for 45° and 57° respectively. Although these absolute values are not particularly useful, the differences between the levels indicate whether the volume will increase, decrease or remain constant as the compression ratio is changed. By averaging A, D and G, it is possible to repeat the procedure for 60lpcm screen rulings. This is also repeated for the other conditions.

0

0

The test image used is shown in Figure 3-2, and by using the arrays, it was possible to reduce the number of gradation scales required from 168 to 52, plus two repeat engravings, and three running across the web. In addition it was possible to reduce the number of experiments required for investigating the screen rulings (3), stylus angles (3) and compression ratios (3) to 19 from 27, a reduction of approximately 30% in measurement time. This cut the number of experiments used from those used in a full factorial experiment by using the reduced experimental set of an orthogonal array, and secondly by 'nesting arrays' – that is, by not repeating identical experiments which appear in multiple arrays. For full details of which conditions were nested, see appendix 2

For the purposes of this investigation, screen ruling (SR), stylus angle (SA) and compression ratio⁵ (CR) were investigated at the following levels.

SR (lpcm ⁶)	SA (°)	CR (°)
60	120	37
70	130	45
80	140	57

Table 3.1 - Variables and their levels

⁵ Compression ratio is measured in degrees, as is the convention in the industry. A better term may be 'cell angle' but compression ratio has been used throughout for consistency and comparison with terms used in industry. It defines an engraving angle

⁶ Screen ruling measurements are given in lines per centimetre (lpcm) rather than the more traditional lines per inch (lpi) as these are the units that most modern engravers work in, including the engravers who produced the cylinders used over the course of the trial.

These variables were chosen because it is these three variables that are selected by the engraver in an attempt to control the engraving. The levels of the variables were chosen to cover the range of conditions that most engravers will use, although under certain conditions, values outside the ranges chosen may be used. This was the purpose of the additional engraving (extending screen ruling down to 50lpcm and up to 100lpcm, to examine the 110° stylus, and to examine compression ratio from 30° to 60° in approximately even steps), however is outside the scope of this particular investigation.

3.2.2.2 Ink release from Non-Image areas

0

0

0

A further three cylinders were produced to investigate ink release from non-image areas (scumming),

Figure 3-6, Figure 3-7. These cylinders consisted of a series of polished bands, with a small engraving in each one. The first two cylinders were produced to investigate the effects of changing ink type (NC or PVB), doctor blade load and substrate type (white OPP, clear OPP, metallized foil) in addition to the surface finish.

Each cylinder was polished on a Daetwyler polishmaster. This produces an extremely smooth cylinder. The centre third was then polished with a stone, and the two outer sixths were then polished with a paper. Once the image was engraved and chromed, one half of the cylinder was polished with a stone, and the other half was polished with a paper, to give six different finishes on each cylinder, as detailed in figure 3.6.

Two different stones (2000 and 3000 grit) and two different papers (9 μ m and 15 μ m) were each used. These values are the two usual stones and papers that are used by engravers in the UK.

42



Figure 3-6 - Cylinder layout

For trial two, where the ink was to be more fully examined (three different types of NC ink), only four surface finishes were used.

r		1	
9µm Paper	2000 Grit stone	2000 Grit stone	15µm Paper
F16	F15	F14	F12

All chrome, 800 grit stone

Figure 3-7 – Cylinder 3 Layout

These finishes were selected to cover the range that was used by the printer that supplied press time. A repeat finish was included as a control factor (the two 2000 grit stone finishes).

3.2.3 Cylinder Measurement Techniques

0

0

As detailed in chapter 2, interferometric analysis was used for quantification of the features of the cylinder. Several other methods were initially considered, and are discussed here. Collaging confocal microscopy was considered, and eliminated due to the lack of quantification available on the results. Atomic force microscopy was

eliminated due to the depth of the cells being larger than the maximum height differences measurable on an atomic force microscope. Scanning electron microscopy was also eliminated due to the need to section the large cylinders, effectively destroying them, and scanning tunnelling microscopy was excluded due to the lengths of time required to measure even a single cell. Traditional methods of cylinder analysis (based on image processing) were used in the setting up on the engraving equipment, but these do not show any detail inside the cell. By assuming cell geometry, this leads to an inference of volume rather than the measurements required. thus the only available method remaining was white light interferometry. This section explains how these measurements were carried out. As the equipment is supplied, it is unable to perform analysis of cell geometries / volumes to the level of accuracy required for this investigation, and as such new techniques and analysis methods had to be defined before this could take place. They are primarily related to the need to measure data inside the cells. The reasons for this are detailed in section 3.2.5. Measurement work was carried out, using two WYKO rollscopes (Figure 3-8), each utilising different internal optics, to encompass the full range of engraving specifications, and subsequent cell geometries. Post processing of the results was carried out to calculate results from the raw data obtained. First the measurement of surface roughness is discussed, as this information is used in the calculation of volume. Following this, a discussion of the calculation of cell volumes is given. Other parameters are also assessed, and the calculation of these is given. All these techniques were performed using VSI measurements (see 2.2.2) due to the increased vertical measurement range necessary for the measurements, and available only in VSI mode.

The filling state of the cells was not quantified, although reference is made to Kunz's work [25]. It was not possible to investigate this due to the nature of industrial materials. Kunz used non-standard inks, and sectioned the cylinder, photographing the cell to see how much ink was inside. This was not possible for a variety of reasons. Firstly, this necessitates the destruction of the cylinder, and secondly, due to the volatility of the solvents used in production inks, it is likely that the ink would have mostly dried within a short period of time, the evaporation significantly affecting the measurements.

0

FAST FELT 2010, pg. 59 Owens Corning v. Fast Felt IPR2015-00650 Finally, it should be noted that this thesis details the first working technique to accurately measure the geometry and volume of a cell.



Figure 3-8 - Rollscope

3.2.4 Surface Roughness

0

G.

Surface roughness measurements were used for the top surface of the printing cylinder. These were required for volume and depth measurements (see 3.2.5 and 3.2.6) and scumming investigations (chapter 5). Historically, profilometers and interferometers have been used to evaluate the roughness of a given surface, and several parameters are available. Analysis of cylinder surface finish was performed using several standard terms; Ra, Rq, Rz, Rt etc. These terms are detailed and defined in [24]. The precise terms used are two-dimensional parameters modified to fit the three-dimensional measurement by addition of a series of two-dimensional measurements. This was carried out internally by the analysis software, and simply uses all data taken in the measurement, rather than a single strip of data. This allows direct comparison between the Ra values calculated on a 3D instrument, such as an interferometer, with a 2D instrument, such as a stylus profiler. Some new instruments use a new term 'Sa' ('Sq', 'Sz' etc) to indicate 3D measurements. These terms include weighting factors and cannot be compared with current 2D measurement results.

FAST FELT 2010 , pg. 60 Owens Corning v. Fast Felt IPR2015-00650