Under the microscope

Visualisation of the distribution of inkjet ink on surface pigmented paper using focused ion beams.

In inkjet printing, it is important to be able to see what happens to the ink on and in the substrate. As this study shows, focused ion beams (FIB) can be used to section through both unprinted and printed inkjet paper. With this technique, the conductive nature of the inkjet pigments makes their distribution after printing easily visible. The high resolution also permits to clearly visualise the pores inside the dried inkjet printed layer.

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The market for inkjet printing has been growing in volume considerably in the last few years and indeed printers in this technology are to be found in most homes and offices. There has also been considerable development in the substrates, the inks and most notably the inkjet printers themselves. One of the key requirements for a good print is that the ink colourant is held near the surface of the coating, without significant spreading. This is needed to obtain a high colour density without wicking and bleeding. There has been a lot of work to improve the properties of the paper substrate to give optimal inkjet printing performance [1]. A wide range of paper grades is available, ranging from polymer-coated plastic substrates, which provide photo-quality prints, to untreated copy paper for the high-volume and low-quality markets. In recent years, many manufacturers have looked at producing a medium-quality paper, using treatment of the surface with pigments. This improves the quality of an uncoated office paper, whilst still maintaining a low cost. However, conventional coating pigments, such as kaolin and calcium carbonate are not often used, as they generally have a low void volume available for fluid uptake [2].

What makes a good inkjet paper?

In general the requirement for a good inkjet paper is good holdout of the ink pigment or dye in order to obtain high colour density without any spreading of the ink, which causes a loss of the definition. The substrate needs to absorb the ink solvent quickly, to stop any lateral spreading of the drops. This will prevent problems such as feathering and ink colours bleeding together. The colourant however has to be retained in the surface layers to ensure a high colour density. In water-based systems this is sometimes achieved by using a cationic coating colour, which helps to hold the anionic ink near the substrate surface. Particles with a high internal pore volume such as silica are often used to produce an internal void structure which promotes rapid removal of the water phase [3]. Other desirable properties include water and light fastness of the paper, stiffness, lack of curl and no strike through of the ink to the reverse side of the paper. According to Svanholm and Strom [1], blurriness and line width are not influenced by the same properties of the coating layer. Line width is related to bulk spreading of the ink before it can be absorbed by the coating. This is controlled by the surface energy of the coating and the ink. Blurriness however is related to the surface micro-roughness characteristics of the coatings.

Techniques to study the distribution of the ink in the paper have been explored by many authors for example Vucak et al [4], Uchimura et al [5] and Glittenberg et al [6].

Focused ion beams as analysis tools

Recently, focused ion beam (FIB) cross-sections have been used to visualise the penetration of ink into the paper. The more conductive nature of the ink used here compared with the coating gave high contrast between them, allowing easy assessment of the penetration. This method has been used to visualise rotogravure, sheet-fed offset and UV-cured offset inks within coated paper surface layers [7], [8]. The preparation of thin sections of paper using the focused ion beam (FIB) technique has been demonstrated by Uchimura et al [9]. These authors showed that it was possible to obtain thin sections of printed paper that could subsequently be analysed by the EDX attachment on a SEM. The paper sample did not suffer from the usual structural changes or artefacts associated with resin embedment. Recently, Uchimura et al [10] have further developed the technique to differentiate between ink pigment and ink resin by first treating the printed paper with osmium tetroxide. This attaches itself to the double bonds in the ink vehicle and allows the detection of the ink vehicle alone by techniques such as EDX. However the osmium tetroxide can also attach itself to double bonds in the latex binder. The ink pigment (barium sulphate) was detected directly by the EDX technique. They showed that in screen printing the ink pigment was held on the surface of the substrate, but that the ink vehicle penetrated into the centre of the paper, through its small pores and along the inside of the fibrils.

The FIB was used by Uchimura et al [5] to prepare thin sections of inkjet papers printed with dye-containing inkjet inks. The distribution of ink was observed using a combination of SEM, optical microscopy and an electron probe micro analyser (EPMA). However the FIB instrument itself was not used to visualise the printed paper. In this work the relatively high spatial resolution of the focused ion beam instrument has been used to give a clear visualisation of the distribution of pigmented inkjet ink after printing.

Materials and methods

The substrates used were a commercially-available, silica-coated inkjet paper and a lightly-pigmented paper, coated with a 3 gm-2 layer of AstraJet 5890. These papers were printed with HP ink using a Hewlett Packard 970 Cxi desktop printer, and the black areas were studied. This ink contains an ink pigment rather than a water-based dye as colourant. An FEI FIB201 gallium focused ion beam instrument was used for sectioning and high-resolution imaging. The instrument is capable of producing a gallium ion beam of between 7nm (at 1pA beam current) and 300nm (at 12nA) in diameter at 30keV energy. A platinum organometallic gas injector allows ion-beam assisted deposition of platinum over selected areas of the sample. This facility was used prior to the sectioning shown here in order to protect the top surface of the sample during ion milling. For sample sectioning, initially a large ion current was used to remove a staircase-shaped trench. A finer beam of lower current was then used to ‘polish’ the larger vertical face of the trench by scanning the beam in a line and moving it progressively up to remove further material. The sample was then tilted to 45° and the polished face imaged using the same ion beam, generally at a much lower beam current to achieve high resolution.

Conductivity issues
Figure 1 shows the geometry of the FIB 'trench'. The images in the results section are taken from the rear wall of this trench, viewed from above at an angle of 45°. In the examples shown in this work, the samples have been cut and polished and then imaged without coating of the polished cut face with a platinum film. It has been found that 'organic rich' components such as the ink absorb the gallium rendering them more conductive, so they are easily imaged in this technique. However the coating layer does not absorb the beam to any great extent and the particles are not imaged without a conductive coating. Hence the ink layer can be seen black, whereas the coating layer is not visible [7]. The images were captured using the ion-induced secondary electrons the energy distribution of which peaks at a few electron volts. If the potential of the sample rises by a few volts, then this signal is severely curtailed and the image will appear dark. For the potential of the sample to rise by a few volts as a result of a 4-10pA ion current impinging upon it, the resistance of the sample to ground must be >10^{12} Ω. It appears that this was the case for the coating material, while for the ink the resistance was less than this, and its appearance was light. After this image has been collected, the cut section is coated with a layer of Pt, which allows visualisation of the particles as well as the ink layer. An example of sectioned and printed paper is given in Figure 2.

Visualising detailed structures

An FIB image of the surface of the silica paper is shown in Figure 3. The platinum strap deposited prior to etching is visible. The very open rough and porous structure of the paper can easily be seen. The silica particles contain micro-voids which can be seen after the FIB sectioning (Figures 4a and 4b).

In Figure 4a, the sectioned silica paper can be seen. The very micro-rough nature of the surface makes it difficult to apply the thin platinum strip evenly prior to cutting. The internal surface of the silica is not conductive and appears black until the section is coated with platinum (Figure 4b).

In Figure 5, a higher magnification image of the sectioned silica particles can be seen. After Pt coating the internal pores are visible. The surface of the printed paper can be seen in Figure 6. The particular nature of the inkjet ink pigment can be seen as a cracked 'filter cake' on top of the paper. There is unevenness in the distribution of the ink pigment, which may lead to some visual mottle. In Figures 7a and b, the section through the printed paper can be seen. Figure 7a shows the situation without a platinum coating over the cut face and Figure 7b with a coating. The ink layers are clearly visible beneath the platinum straps, and the contrast between the ink and coating is very high. The extent of ink penetration into the coatings is easy to see, although for statistical reasons it would be necessary to section many regions in order to quantify this.

In Figure 7b, application of the platinum allows the paper fibres and silica particles to be seen. Figure 8 is a high magnification image of the ink film applied to a surface pigmented paper. The high resolution allows the pores inside the dried ink film to be visualised.

References


Results at a glance

- FIB sectioning and imaging were used to study the penetration of inkjet inks into paper coatings.
- It could be seen that the inkjet ink pigment was held out very well along the surface of the pigmented or silica coated paper.
- The conductive nature of the ink pigment allowed its visualisation, and excellent contrast, without a coating of platinum.
- The high resolution of the technique allowed the very small pores within the ink pigment filter cake to be observed.
- The technique of FIB sectioning and imaging is a useful tool in developing substrates and inks for the printing industry.
- Its use can help corroborate existing methods for quantifying ink penetration and distribution, for example print density and print-through analysis.

The authors:

- Janet Preston joined English China Clays Int. (now Imerys Minerals) in 1985 and has worked in the areas of coatings research, both in the area of paints and paper coating and in printing science. Whilst working at Imerys, she graduated from University of Plymouth with an honours degree in Applied Chemistry. She then gained a PhD in Chemistry with the Interface Analysis Centre, University of Bristol, where she studied ink and coated paper interactions. She is currently a scientist in the company’s Paper Technology Group and is working in the area of printing fundamentals. In 2007, she was appointed as an Adjunct Professor at Abo Akademi University in Finland, in the Laboratory of paper coating and converting.
- Dr. Peter Heard took his first degree in Electrical Sciences at the University of Cambridge in 1981 and a doctorate in 1984; his PhD being the first in the area of focused ion beams from liquid-metal ion sources. He has since worked in UK industry for 10 years in the areas of lithography, micro-machining, surface analysis and plasma physics for semiconductor applications. He is currently a Research Fellow at the Interface Analysis Centre, University of Bristol and his present research includes SIMS analysis.
of novel materials and the modification of devices using focused ion beams.

Björn Lindqvist joined Imerys Minerals in 1998, and currently holds the position of Technical Manager Board and Packaging Speciality Papers. He holds a Masters degree in Chemical Engineering (1993) from Abo Akademi University Finland.

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Figure 1: FIB methodology for sectioning printed paper samples
FIB  Sectioned paper - an example

Figure 2: An example of a sectioned printed paper
Figure 3: Secondary electron image of the surface of the silica coated inkjet paper, showing the macro-pores between the particles. Pt strip applied to the surface prior to sectioning.
Figure 4: Section through silica coating. a) The surface of the paper was Pt coated, but not the sectioned face. The internal silica particle voids are not visible. b) Same section coated with Pt, showing the silica particles.
Figure 5: Nano pores are visible inside the silica particles on the HP Premium paper
Figure 6: Surface image of the black printed HP paper. The surface roughness and cracked nature of the ink film are both apparent.
Figure 7: a) The inkjet pigment is easily seen as a discrete layer on the surface of the paper; b) Fibres and silica particles now visible
Figure 8: A high resolution image of an inkjet printed paper shows the pores within the dried ink film.