Making negatives and plates for printing by electroerosion: III. Use of the direct negative and direct plate

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Issues related to the practical usage in the pressroom of both the direct negative and direct plate are discussed. Two concerns associated with the usage of the *direct negative* during platemaking are treated: 1) the effect of light transmission through defects (voids) in the aluminum film, and 2) the effect of light transmission directly through the aluminum. These concerns may be addressed respectively by careful fabrication of the material and careful control of the exposure conditions during platemaking. Two aspects of the usage of the *direct plate* are considered: 1) the need for a simple prepress "activation" treatment, and 2) press-life limitations caused by wear of the direct plate on the press. Passing a direct plate through an activator solution immediately prior to mounting it on the press prevented scumming in the image area and blinding in the background (nonimage) area. The life on the press of the direct plate is limited by the wearing away of the aluminum from the background areas, thereby causing scumming. Changes in the structure of the direct plate which could prolong press life were investigated.

1. Introduction

The physical principles of the direct negative (DN) and direct plate (DP) written by electroerosion on the IBM 4250 printer were presented in Part I of the present series of papers. In Part II issues involved in larger-scale fabrication of the DNP material were discussed. The present paper, Part III, is devoted to a discussion of questions associated with the practical use in the pressroom of this material as a direct negative (Section 2) and as a direct plate (Section 3). The studies described below provided a more complete understanding of such important topics as the exposure process for platemaking with the *direct negative*, and the life on the press of the *direct plate*, and led to techniques which offer improved performance.

2. Usage of the direct negative

• *Background*

The preparation of a typical "negative working" offset plate from a *conventional* silver-halide negative made by the photographic process involves two steps [1]. First, the negative is placed in intimate contact with the photosensitive plate surface in an exposure apparatus (platemaker), and the sandwich is given a controlled exposure to light. Next, the plate is subjected to wet-chemical development. The *direct negative* is created by electroerosion writing of the special DNP material on the IBM 4250 printer. The conventional offset-plate preparation procedure may subsequently be followed using standard plates, but now with *direct negatives* instead of conventional silver-halide negatives. The only modification of the IBM 4250 printer procedures described previously was a software change which was instituted to permit the production of *wrong*-reading direct negatives. This change allowed the direct negative to be placed on a conventional plate with the *aluminum* side adjacent to the plate surface (**Figure 1**). This step was necessary to achieve good resolution. (If the aluminum were separated from the plate by the thickness of the polyester substrate, resolution loss would be caused by penumbral effects.)

The "optimized" DNP material described previously (Part II) does succeed in fulfilling the requirements of operation as a direct negative. Nonetheless, the studies described in this paper have emphasized the need for particular care in control of certain material characteristics, and have shown that some special precautions should be taken when using the direct negative. In particular, two major potential problems associated with the direct negative have been identified which could result in undesired exposure of portions of the plate in the background (nonimage) area: 1) undesired light transmission through defects in the aluminum, and 2) excessive light transmission directly through defect-free aluminum.

• *Defects in the aluminum film*

Reproduction on the plate of light-transmitting defects (voids) in the aluminum film was of rare enough occurrence that the opaquing operation commonly used to mask out defects in conventional silver-halide negatives

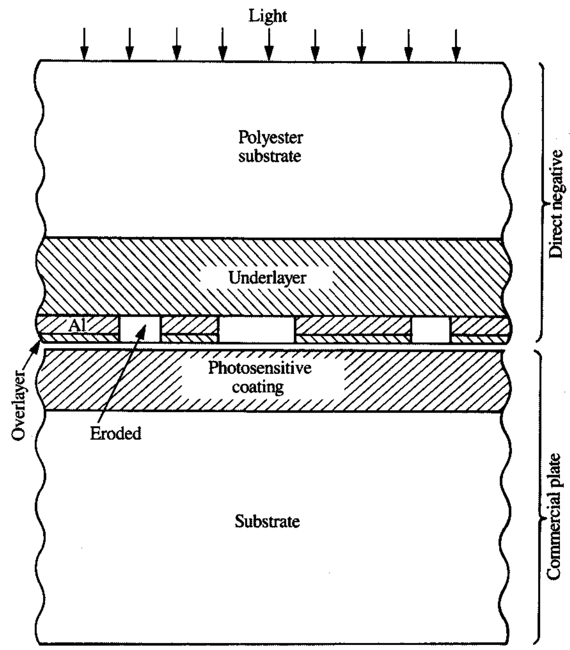


Figure 1

Direct negative in contact with commercial plate during exposure operation. (Not to scale.)

required relatively infrequent application for the direct negative. It was nonetheless necessary to ensure the suppression of such defects,[1] which were of three types:

1. Scratching caused by the writing operation (Part II).
2. Defects induced during fabrication of the DNP material.
3. Defects caused by handling the DNP material.

Scratching-induced defects

It was shown previously that scratching which occurs during writing is associated with the production of a multitude of small holes in the aluminum along the track of the writing head (Parts I and II). If the concentration and size of these holes were large enough,[2] it was found that sufficient light could be transmitted through them during the platemaking process so that the plate was locally exposed, and corresponding small ink dots could be seen on the printed paper. It was noted that this effect became more pronounced as the scratching increased [as measured

[1] Suppression of such defects is also important for the direct plate (Section 2).
[2] Holes smaller than about 10 μm in diameter were found not to reproduce upon exposure and development of the plate.

Table 1 Overexposure through scratching-induced holes: Dependency of maximum permissible exposure-guide step on aluminum OD, ΔOD.

ΔOD	*Aluminum OD*		
	1.6	*1.9*	*2.1*
0.01–0.02	5	6	7
0.05–0.07	4	5	6
0.10–0.12	3	4	5
0.17–0.20	2	3	4

Table 2 Overexposure through scratching-induced holes: Dependency of T_{holes} (%) on aluminum OD, ΔOD.

ΔOD	*Aluminum OD*		
	1.6	*1.9*	*2.1*
0.01–0.02	0.088	0.044	0.028
0.05–0.07	0.37	0.19	0.12
0.10–0.12	0.72	0.36	0.23
0.17–0.20	1.33	0.67	0.42

(Part II) by the optical density[3] difference ΔOD], as the aluminum thickness decreased [as measured (Part II) by the optical density of the background area OD], and as the plate exposure time increased.

To examine this phenomenon more closely, a set of twelve written samples of DNP material was studied, each with differing values[4] of OD or ΔOD. For each sample a series of plates[5] was exposed in a platemaker[6] for various times, then developed. Each plate was then used to print paper which was examined under a magnifying glass for the presence of ink dots associated with scratching-induced holes. For each DNP sample, the maximum exposure time was identified which did *not* produce ink dots.

The results (**Table 1**) have been translated from exposure times to "sensitivity-guide" or "gray-scale" readings, which are commonly used in the printing industry for determining the proper exposure of a plate. A sensitivity guide (for example, the Kodak Control Scale T-14, Eastman Kodak Co., Rochester, NY) consists of a series of standardized steps of increasing optical density. The guide is placed on the plate during exposure. After development (and printing), the areas corresponding to the lower-numbered steps, i.e., steps having lower values of optical density, become visible on the plate (and on the printed page); a longer exposure time causes development of additional steps corresponding to higher optical density. The maximum guide-step number not associated with the presence of ink dots is thus given in Table 1.

It is seen from Table 1 that for DNP material of a given aluminum thickness (OD), as the value of ΔOD increases, the maximum permissible guide-step number decreases. It is also seen that for a given value of ΔOD, as the aluminum thickness (OD) increases, the maximum permissible guide-step number increases. It is further noted that for optimized DNP material (OD of about 2, showing scratching such that ΔOD is less than about 0.1), the manufacturer's recommendation of exposure to step 6 is nearly met; an exposure to step 5 would be preferred. However, it should be observed that excessively pronounced underexposure is not a practical solution to this problem because such underexposure could lead to shortened life of the plate on the press. (When a conventional plate is considerably underexposed, the light-induced cross-linking of the coating in the image area is inadequate. The action of the press then causes this material to wear away prematurely, so that the image area fails to pick up an adequate amount of ink. The plate then goes "blind.")

The relationship among aluminum thickness, scratching, and maximum guide-step number shown in Table 1 may be clarified by an examination of **Table 2**, where the value of T_{holes} is given for each sample of DNP material. The latter parameter, which represents the light fraction which would be transmitted only through the scratching-induced holes, is equivalent to the areal fraction of holes in the scratched region (Part II). Here the relation

$$T_{holes} = 10^{-OD}[10^{\Delta OD} - 1]$$

was used to calculate T_{holes} (Part II). By comparing Tables 1 and 2 it is seen that, as expected, the determining parameter here is indeed T_{holes}, which increases with ΔOD for constant OD, or with decreasing OD for constant ΔOD.

In summary, the scratching did not cause unacceptable results if the DNP material was made according to the procedures outlined previously (Part II), if the electroerosion writing was properly performed, and if a sensitivity guide was used to keep the exposure during platemaking at or slightly less than the manufacturer's specifications.

Fabrication-induced defects

Fabrication-induced defects took one of two forms: long narrow scratches in the machine direction and scattered pinholes.

Machine-direction scratches were found to originate during the overlayer coating operation, and could be minimized by careful attention to the overlayer coating-fluid preparation and coating procedures (Part II). Any

[3] White-light optical density was measured for a wide-wavelength band by a laboratory transmission densitometer. The instrument used was the TD904, Macbeth Div., Kollmorgen Corp., Newburgh, NY.
[4] To obtain different values of ΔOD for a given aluminum thickness, DNP samples of different roughness and overlayer thickness were chosen.
[5] 3M Corp., St. Paul, MN.
[6] "Sun," PRT Co., Milan, Italy.

remaining scratches were usually so narrow (<10 μm wide) that they did not reproduce on the plate.

Many of the pinholes associated with the fabrication process were also so small (<10 μm) that they did not reproduce on the plate, but there were occasional large pinholes which did reproduce. Microscopic examination revealed two types of pinholes. In the first, the aluminum was locally removed but the surface of the underlayer was undisturbed. In the second, not only the aluminum film but also portions of the underlayer were locally lacking.

Three different origins of pinholes have been postulated:

1. The coating and calendering operations caused buildup of a high-density electrostatic charge on the underlayer surface, so that dust particles from the air were attracted to this surface and held by electrostatic forces. These dust particles caused pinholes by shadowing the metal vapor during deposition [2]. Also, the deposited metal dissipated the electrostatic charge of the dust particles so that they could easily be removed from the substrate during subsequent handling. Since the dust particles carried deposited aluminum with them, pinholes were thereby created.
2. During calendering, microscopic portions of the underlayer were found to tear off and adhere to the steel calender rollers, as could be seen by examination of the surface of these rollers. Some of these transferred "pull-out" particles were redeposited on the underlayer in subsequent revolutions of the calender. These particles had poor adhesion, and when they left the material with their deposited aluminum, pinholes were created.
3. When fragments of the underlayer were pulled out by the calendering process, holes were often left which were narrow and deep. The aluminum deposits in the bottom of such pull-out holes were found to be considerably thinner than normal because of the shadowing effect of the high walls of the holes.

From these considerations it is clear that careful attention to clean-room techniques, along with electrostatic charge removal by deionizing apparatus during the coating and calendering operations, should help to minimize dust-induced pinholes. The calender-caused pull-out problem may be ameliorated by attention to calendering procedures. (In particular, the calender rollers should be in contact with the underlayer for the minimum time so that fragments of the underlayer do not overheat and transfer to the calender rollers. Particular care must thus be paid to calender start-up and roll-splicing operations.) In addition, there was some evidence that the use of thicker underlayers helped minimize pinhole concentration; for example, an increase in underlayer thickness from 5.0 to 5.9 μm caused a factor of 3 decrease in pinhole density. Such thicker underlayers may be less subject to the calender pull-out effect.

Careful exposure control during platemaking should also be beneficial in minimizing exposure through residual fabrication-induced defects.

Handling-induced defects

The major defects associated with handling of the DNP material are holes in the aluminum film originating in corrosion of areas previously contacted by the fingers. It has been found that in the presence of a suitable electrolyte, such as that present on human skin, the aluminum film and the graphite of the overlayer can function as an electrolytic cell, thereby causing the corrosion. This problem can be minimized by careful handling so as to avoid finger contact with those regions of the DNP material directly corresponding to the pages being printed.

- *Exposure through aluminum*

The optical density of a conventional silver-halide negative can be 4 or higher, so that negligible light passes through the background area of the negative even for the longest overexposure in the platemaker. In contrast, as discussed previously (Part II), the optical density of correctly fabricated DNP material should be no greater than about 2, so that in this case a long overexposure *could* allow some unwanted exposure of the background area of the plate, causing the press to print corresponding gray regions, i.e., to "scum" in the background area.

The background overexposure problem can in principle be attacked by increasing the optical density by increasing the aluminum thickness. However, as shown previously (Part II), the practical upper bound for the optical density of the aluminum is about 2, because for thicker aluminum the writing quality deteriorates.

The solution to this problem again lies in careful exposure control with a sensitivity guide, so that the plate is not overexposed and is perhaps slightly underexposed. Exposure test results are given in **Table 3** for three different commercially available plates: two "additive" diazo-based plates and a "subtractive" photopolymer-based plate. For an additive plate a hydrophobic polymer or lacquer is applied as one of the development steps; such a polymer preferentially adheres to the image area to provide increased press life. The hydrophobic polymer is already present in a subtractive plate and is removed from the background areas during development. The chemistries of the diazo-based and photopolymer-based plates are thus different.

These plates were used in conjunction with two different unwritten DNP samples of different optical densities. (Optical densities below the standard value of 2 were deliberately chosen to provide a worst-case test.) The

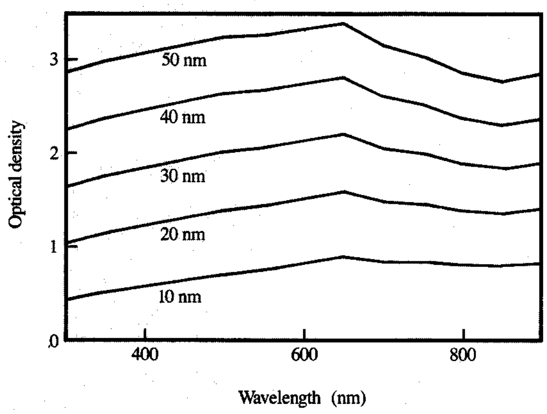


Figure 2

Computer-derived plots of optical density vs. wavelength of aluminum films of various thicknesses on a polymer substrate.

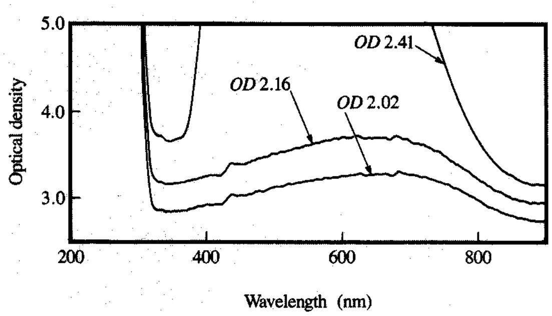


Figure 3

Spectrophotometer traces giving optical density vs. wavelength of aluminum films of various thicknesses as indexed by their white-light optical density.

setting which controlled the light exposure in the platemaker[7] is given in arbitrary units in Table 3 along with the corresponding highest guide-step number which was fully developed. In each case the guide step recommended by the plate manufacturer was step 6.

These results indicate that in all cases the manufacturer's recommended guide step could be achieved or sometimes exceeded without the appearance of any scumming in the background area when the developed plates were tried on the press. The only difficulties encountered were associated with exposures corresponding to step 7 or 8 using the 3M Type E diazo-based plates (this case is not shown in Table 3); such an exposure exceeded the manufacturer's recommendations. On the other hand, photopolymer plates, which are increasingly used in preference to diazo-based plates, could be subjected to an exposure considerably beyond the manufacturer's recommendation without background area scumming.

[7] The platemaker (NuArc Co., Chicago, IL) used a mercury arc light source.

Table 3 Overexposure through aluminum.

Commercial plate	*Plate type*	*DN code number*	*Al OD*	*Exposure setting*	*Developed guide step*
3M: Type E	Add Diazo	B103	1.89	16	2.5–3.5
				24	3.5–5.0
				36	5.0–6.0
		30A8	1.99	16	2.5–3.5
				24	3.5–5.0
				36	5.0–6.5
3M: Type R	Add Diazo	B103	1.89	16	3.5
				24	3.5
				36	6.0
		30A8	1.99	16	3.0
				24	4.0
				36	6.0
Kodak: SX	Sub Photo-polymer	B103	1.89	16	8.0
				24	8.0
				36	9.5
		30A8	1.99	16	7.0
				24	8.0
				36	10.0

1. Add = additive plate: Lacquer is added in development to improve press life; Sub = subtractive plate: Components are removed during development.
2. Exposure time measured in arbitrary units on platemaker using mercury arc.
3. Partial step development reported as fraction: for example, 3.5 means solid step 3 and partial step 4. When the same exposure was given to three or four identical plates, variability in sensitivity-guide results was found; in these cases the guide-step results are reported as a range.
4. None of the plates tested for the exposures shown scummed in the background area when tried on the press.
5. 3M Type E and Type R plates from the 3M Corp., St. Paul, MN. Kodak SX plates from Eastman Kodak Co., Rochester, NY.

- *Optical filters to inhibit overexposure*

The studies of exposure through defects or directly through the aluminum show that in both cases overexposure effects can be prevented by careful control of the exposure. However, it must also be noted that there is a large variety of offset-lithographic plates on the market, all of which have different exposure and development characteristics. Furthermore, these characteristics are dependent upon the spectral characteristics of the particular light source used, and upon the development technique employed. In order to avoid overexposure effects it is necessary to choose the plate, the light source, and the development technique with care. In some cases it may be necessary to use an *under*exposure of perhaps two or even three steps below the plate manufacturer's recommended exposure guide number, with the acceptance of some loss of press life.

Fortunately, it has been found that more tolerance in the exposure time can be obtained by the installation of appropriate optical filtering in the exposure system. To develop this approach, a study was undertaken of the spectrophotometric characteristics of the total exposure system consisting of light source, DNP material, and conventional plate.

As a first step, calculations were made of the net optical density of aluminum films of various thicknesses on polymer substrates as a function of illumination wavelength. Handbook values of the complex refractive index were used in these calculations, and computer-derived plots were generated (**Figure 2**). It may be seen from these curves that the optical density decreases for shorter wavelengths in the ultraviolet. Experimental confirmation of this trend was obtained from spectrophotometer traces of DNP material of various aluminum thicknesses, as indexed by their white-light *OD* values. The results[8] are shown in **Figure 3**. (The sharp increase in optical density below 310 nm is caused by the strong ultraviolet light absorption of the polyester substrate.)

The net spectral sensitivity of the total system of light source, direct negative, and commercial plate is given in principle by the convolution of the curves associated with each of the three components. Light sources commonly used with commercial platemakers include carbon arc, mercury arc, pulsed xenon, and metal-halide sources, each having different spectral characteristics. While these spectral characteristics are available, it is unfortunately difficult to obtain the spectral sensitivities of commercial plates, so that it is impractical to perform the desired convolution. Nevertheless, it seems to be generally recognized that diazo plates show a peak in sensitivity at about 370 nm, while photopolymer plates usually have a peak sensitivity above about 410 nm. From these considerations it is seen that use of light sources rich in the shorter wavelengths (e.g., a carbon arc), and/or use of those commercial photosensitive materials more sensitive to the shorter wavelengths [e.g., diazo plates or "Dylux" proofing material (E. I. du Pont de Nemours & Co., Wilmington, DE)] can lead to more pronounced background scumming due to transmission through the aluminum. These expectations have been verified by experimental evidence. (As one example, Tables 1 and 2 show that certain photopolymer plates are less prone to background overexposure than are diazo plates.)

By taking into account the spectral characteristics of the light source, the plate, and the DNP material (Figure 3), it would seem possible to extend the exposure tolerance limits by inclusion of an optical filter which blocks the transmission of the shorter wavelengths. Under these circumstances the aluminum film of the DNP sample would be made effectively more opaque to the incident radiation, as desired. The cutoff wavelength of such a filter must be chosen with some care, however. If the cutoff wavelength is chosen to be considerably less than the peak spectral sensitivity of the plate, the filter will be ineffective. On the other hand, if the cutoff wavelength is chosen to be considerably higher than the peak spectral sensitivity of the plate, the background overexposure problem will be helped, but at the expense of an unacceptable prolongation of the required exposure time. These concepts were explored in special tests.

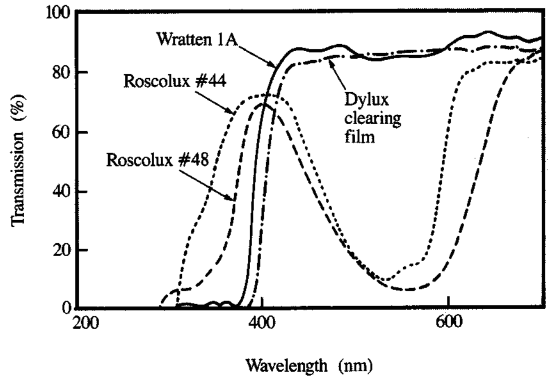
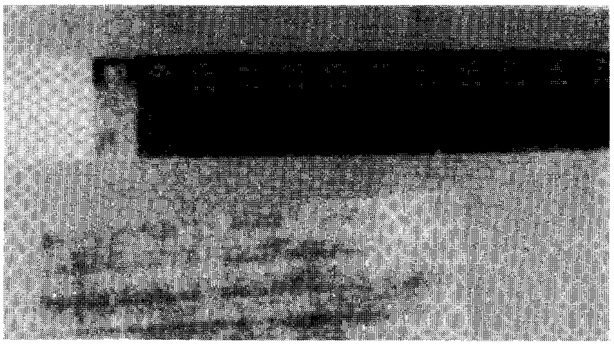


Figure 4

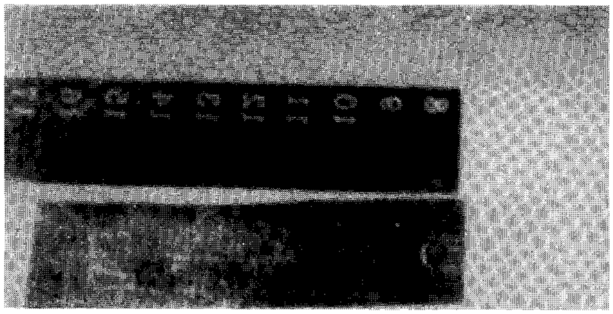
Spectrophotometer traces giving optical transmission vs. wavelength of various filters.

It was most convenient to have such a filter in the form of a thin, flexible foil, which could either be placed over the sandwich of the direct negative and the commercial plate, or alternatively be positioned in front of the light source of the platemaker. Spectrophotometer traces were made of several potentially useful filter foils (**Figure 4**). It was observed that many commonly available filters had an identical cutoff wavelength of about 400 nm. However, a useful category of inexpensive filter foils of different spectral characteristics was found in filters customarily used for colored stage lighting (Rosco Corp., Port Chester, NY). The cutoff wavelength of Roscolux 44 was relatively short, that of Roscolux 48 was intermediate, and that of Roscolux 49 (not shown in Figure 4) was highest. Simple tests were carried out with DNP material of very low optical density (1.7) using each of these foils in two platemakers, one with a metal-halide light source and the

[8] The curves corresponding to the higher optical densities are in error because of instrumentation failure at very low light levels.



(a)



(b)

Figure 5

Effectiveness of filtering on background overexposure effect. A sensitivity guide, together with a coupon of DN material of 1.7 optical density, was exposed to a sensitivity guide step of 7. A black region corresponds to the unexposed state, while whitening of a region signifies exposure of the photosensitive plate material: (a) No filter was used. (b) A Roscolux 49 filter covered both the DN coupon and the sensitivity guide.

other with a mercury light source. Since the diazo plate was more critical, the 3M Type E plate was used for these tests.

The results showed the most decrease in background scumming for long exposures with a 400-nm cutoff filter and significant improvement with a Roscolux 48 filter, but relatively little improvement with a Roscolux 44 filter. On the other hand, a pronounced increase in the required exposure time was needed when a 400-nm cutoff filter was used. It would therefore seem that a filter having the characteristics of the Roscolux 48 (cutoff roughly 350 nm) or the Roscolux 49 filter (slightly higher wavelength cutoff) is a good compromise.

The filter and no-filter cases are compared in **Figure 5**. Here a sensitivity guide and a coupon of DN material of only 1.7 optical density were exposed together, using 3M Type E plates with a platemaker having a metal-halide light source. A black region corresponds to the unexposed state, while whitening of a region signifies exposure of the photosensitive plate material. [The original red (exposed) and silvery (unexposed) colors of the developed plate were translated to white and black, respectively, by a color-separation process. J. C. Lee is thanked for making this translation.] In Figure 5(a) no filter was used, while in Figure 5(b) a Roscolux 49 filter covered both the DN coupon and the sensitivity guide. In both cases an exposure corresponding to a guide step of about 7 was given; exposure time for Figure 5(b) was more than three times longer than for Figure 5(a). It is seen that while the region corresponding to the DN coupon is nearly all white (exposed) in the no-filter case [Figure 5(a)], it is mostly black (unexposed) in the filter case [Figure 5(b)].

It was found in practice that the use of such filters permitted an increase in exposure corresponding to one or two guide steps higher than possible without filtering. Even though the use of these filters necessitates an increase of exposure time over the no-filter case by a factor of 2 to 3, in many instances it may be advantageous to use a filter in order to widen the permissible exposure latitude.

3. Usage of the direct plate

• *Background*

The usage of the direct plate is straightforward in principle, since it involves writing on the IBM 4250 printer, then mounting the written sheet on the press and operating the press in the normal fashion. However, further study showed that certain topics deserved closer attention.

1. The effect of defects in the aluminum film.
2. The need for a simple prepress "activation" treatment.
3. The mechanism of wear on the press, which limited press life.

Testing of the direct plate was carried out on two small "duplicator" offset-lithographic presses, each operating on a different ink and water supply-system principle [1]. The first[9] had a "conventional" dampening system which involved two separate trains for the water and ink, while the second[10] employed a form of the "integrated" (Dahlgren) dampening system which uses a single form-roller for application of both ink and water to the plate. For operation with either press, a written sheet was removed from the IBM 4250 printer, cut to the correct size, and punched with a series of holes at the top and bottom edges for proper gripping to the plate cylinder,[11] then mounted for normal press operation. Tests with both presses gave similar results, which were satisfactory in general.

Since the topics of fabrication-induced defects and writing-induced defects (scratching) were treated

[9] AM Multigraphics 1250, AM International, Chicago, IL.
[10] Most of the testing with an integrated-dampening press employed a TOK press, Heidelberger Druckmaschinen Ag., Heidelberg, Germany. Some testing was also carried out on a 360 CD press, A. B. Dick Co. Inc., Chicago, IL.
[11] The "pin-bar" clamping system was used. Unless otherwise noted, standard electrophotographic copy paper or offset-lithography paper was used in this work.

previously (Parts I and II), little discussion is needed here. Such defects are undesirable for the direct plate because they represent ink-receptive areas. As in the direct-negative case, it was found that fabrication-induced defects less than about 10 μm in width did not reproduce with a direct plate, at least for the initial press copies. For this reason, neither fabrication-induced defects nor writing-induced defects normally presented a problem for the direct plate if the DNP material was fabricated according to specifications and if the electroerosion writing was carried out properly. However, as explained subsequently, if these conditions were not fulfilled the resulting defects caused shortening of the press life.

• *Prepress activation treatment*

Before running the press it is common practice to pretreat or wash a conventional plate, sometimes with special chemicals. In the present case it was found that the direct plate also needs a prepress "activation" for acceptable results. If a direct plate was mounted and run on the press *without* activation two problems were encountered: 1) The *background* (nonimage) areas were often gray, since they took some ink, i.e., were "scummed." 2) The *image* areas appeared grayish instead of black because they had inadequate ink uptake, i.e., were "blind." These problems were most pronounced for the first few copies from a fresh plate, but as the press ran the copies improved in quality; i.e., the background areas became cleaner, while the image areas became blacker. Simply running on the press was not a practical solution, however, since typically many hundreds of copies were required before acceptable results were achieved.

Scumming in background area

The scumming is caused by inadequate hydrophilicity of portions of the background area. This lack of hydrophilicity of the direct-plate surface is probably caused by adsorption of hydrophobic contaminants from the air in the time since fabrication of the DNP material. Such increase of hydrophobicity is a common phenomenon [3]. For example, a freshly deposited aluminum film is hydrophilic, with a contact angle with water approaching 0°, but after exposure to the air for about a day the contact angle increases to over 30°. It may also be noted that a freshly developed *conventional* lithographic plate also becomes hydrophobic upon exposure to the atmosphere for several hours. (If use of the plate on the press is delayed, such a plate should be protected with a coating of a hydrophilic water-soluble gum such as gum arabic.) The cited monotonic decrease in scumming with continued running of the direct plate on the press may be explained by the repeated application on the press of fountain solution which serves to clean the background area and render it hydrophilic.

It was found that if care was taken to clean the surface of the direct plate in a special way immediately before mounting on the press, no scumming was observed. The cleaning apparently removed any adsorbed hydrophobic film. Cleaning solutions involving detergents were tried for this purpose, but it was found that a very light etching action[12] with either acid or basic solutions applied by means of an absorbent pad was most satisfactory, and that a light rubbing action during application was beneficial. An effective alternative cleaning procedure was found to be exposure to a plasma in air at reduced pressure for a few seconds in a "plasma-ashing" apparatus (LFE Corp., Clinton, MA). However, this treatment was impractical because it did not aid in ink uptake, and in any case would be cumbersome for routine operation in printing. Nonetheless, this treatment was useful for press tests of coupon samples.

Blinding in image area

The parallel problem of inadequate ink uptake in the image areas may be explained by inadequate hydrophobicity of those regions. This inadequate hydrophobicity is caused by residual hydrophilic fragments of aluminum which remain after the electroerosion writing process. The improvement in ink uptake as the press continued to run may be explained by the wearing away of these residual fragments by the press action. In a simple test of this explanation, the press-wear mechanism was simulated by the strip-off of some of the aluminum fragments in the image area by means of pressure-sensitive tape; the image areas so treated exhibited very good ink uptake when the press was started.

Further study showed that the prepress application of a mild etchant to the direct plate yielded excellent ink uptake. A reproduction of a portion of one of the first few copies of a page printed from an etchant-treated direct plate is shown in **Figure 6**. Using an absorbent pad, a strong (pH = 3) phosphoric acid etchant was applied to the top left portion of the area, a weaker (pH = 4) phosphoric acid etchant was applied to the top right, and no etchant at all was applied to the lower portion of the area. It is seen that the stronger etchant gave the best results; with no etching only a very weak gray image is seen.

The edge of an electroeroded region is very ragged; as this region grows during erosion, "peninsulas" of aluminum are sometimes cut off so that residual islands are formed which can vary in size up to tens of μm in width. (The total area of these residual islands is only about 5% of the eroded region.) It was thought at first that these islands were responsible for the undesired hydrophilicity in the written areas. However, examination by optical

[12] The use of such an etching treatment could not, however, effectively eliminate scumming for a DNP having an overlayer incorporating a *hydrophobic* binder, e.g., CAB. This is one of the major reasons why an overlayer incorporating a *hydrophilic* binder, e.g., HEC, was chosen (Part II).

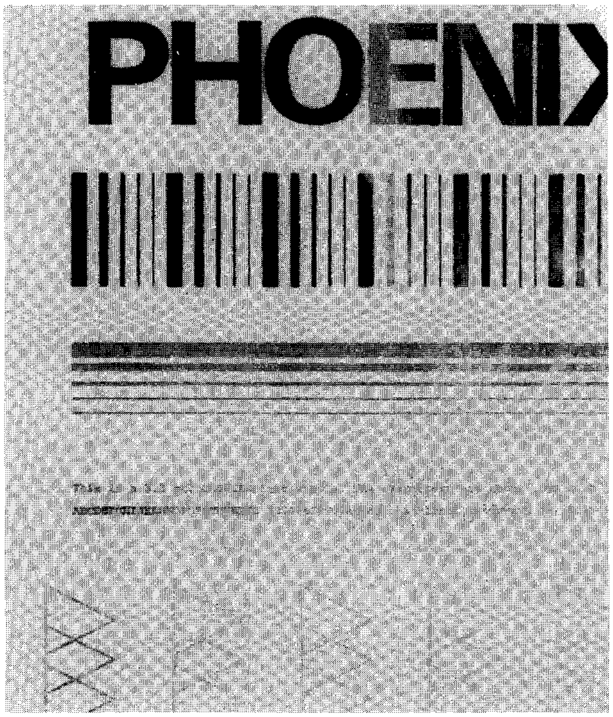


Figure 6

A portion of one of the first copies from the press using a direct plate. Lower region, no activator. Upper left region, manually applied phosphoric acid, pH = 3.0; upper right region, manually applied phosphoric acid, pH = 4.0.

microscopy of written regions before and after an etch which was adequate to give full ink uptake showed very little change in the size of such islands; correspondingly, there was negligible change in the transmission optical density. On the other hand, further study by scanning electron microscopy showed the presence of *globules* of aluminum ranging up to several μm in width and height [**Figure 7(a)**]; these globules were apparently scattered over the surface during the electroerosion process (Part I), perhaps by an "explosive molten-metal release" mechanism [4]. Inductively coupled plasma-emission spectroscopy revealed that the globules accounted for a surprisingly high fraction of the original uneroded aluminum, i.e., nearly 2/3 by volume. The activation process apparently removes the globules in preference to the residual aluminum islands [**Figure 7(b)**], thereby reducing the fraction of aluminum to 1/3 of the original, again as measured by plasma-emission spectroscopy. The removal of these hydrophilic globules by activation is adequate to render the written area hydrophobic enough to accept ink.

Light rubbing during application of the activator solution was found very beneficial in eliminating blinding. It is thought that the globules are probably not simply dissolved by the activator, but rather that the activator solution may help free the globules from the surface so that they are mechanically removable by rubbing (i.e., the activator solution acts as a releasing agent). This would explain why the globules are removed during activation in preference to the larger but much thinner residual islands. Further support of this viewpoint is given by experiments which showed good activation with an activator solution consisting of a mixture of NaF and Na_2PO_4 adjusted to have a pH of 6.5. Such a neutral solution cannot etch *aluminum*, but is known to attack aluminum *oxide*, so that the oxide shell surrounding the aluminum globules[13] was

[13] X-ray photoelectron spectroscopy (XPS) showed that while these globules contained a core of metallic aluminum, they had an outer shell of aluminum oxide.

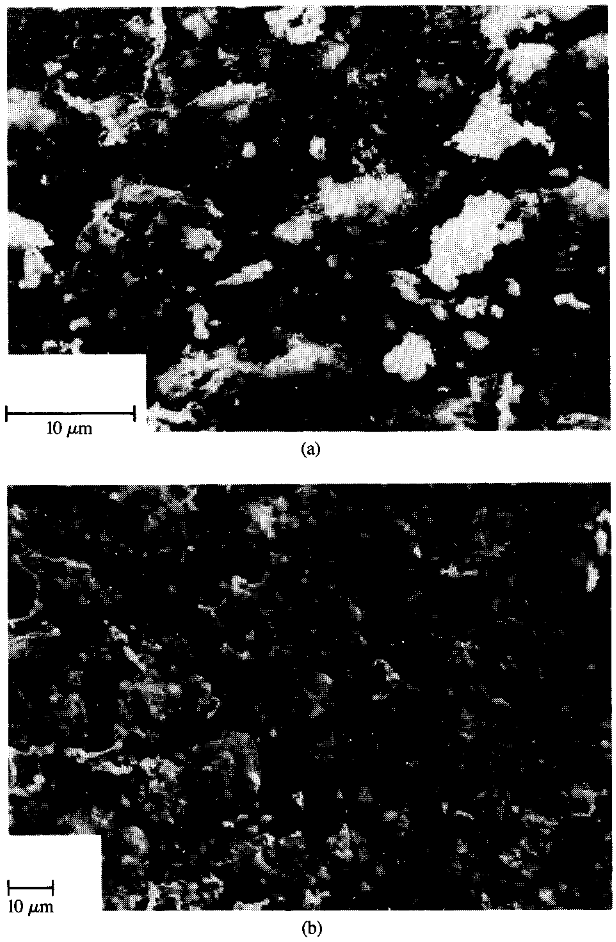


Figure 7

Scanning electron micrographs of an area which had been written on the IBM 4250 printer: (a) No activation given. (b) After activation treatment.

preferentially attacked, thereby releasing the globules so that they could subsequently be removed by mechanical action.

A search for treatments alternative to etching showed good ink uptake upon exposure to other reagents such as long-chain fatty acids (e.g., myristic acid dissolved in ethanol), or water-based emulsions of hydrophobic particles (e.g., hydrocarbon waxes) or liquid hydrocarbons (e.g., mineral oil, butyl stearate, or glycerol trioleate). By means of these treatments the image area became highly hydrophobic. The adhesion of the applied hydrophobic material was good enough to withstand the wear of the press, so that the consequent ink uptake was satisfactory. In contrast, the adhesion of such hydrophobic material to the *non*-image area was poor, so that it did not promote scumming; on the other hand, neither did it do anything to suppress the scumming already present.

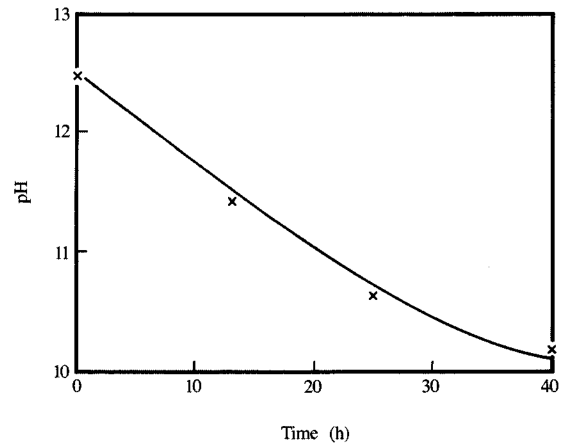


Figure 8

pH of an activator as a function of time: 3M Onyx developer, diluted 10:1. Solution was exposed to air in an open tank. This activator was effective for about 24 h air exposure.

Choice of activator and applicator

After considering the alternatives, it became clear that prepress activation of the direct plate by etching was preferable to other treatments because it allowed two goals to be achieved in one simple treatment: It provided background area cleaning (preventing scumming), while permitting good ink uptake in the image area (preventing blinding). With such an activator, even the first copy off the press was of good quality. However, it quickly became apparent that excessive skill was required in the application of these activators by hand because of the difficulty of avoiding local under-etching or over-etching. (Under-etching could cause scumming or blinding; over-etching could cause loss of press life because of holes etched in the aluminum.) For this reason it was desirable to make the activator application semiautomatic.

This goal was achieved by use of commercially available machines designed either for semiautomatic development of "diffusion transfer" photographic material or for development of "direct image" offset lithographic plates. (Acceptable machines were the AP 15, Processor Corporation of America, Reston, VA, and the DT-15BM-1, Arkay Corp., Milwaukee, WI.) The machines consist of motor-driven input and output roller pairs with an intervening open tank containing the bath. In operation a piece of DNP material was inserted into the input rollers; the sheet was then automatically directed under the surface of the bath and out through the output rollers, which subjected the sheet to the desired mechanical (squeegee) action. With the use of such a machine, uniform application of the activator over the surface of the direct plate was achieved, while the immersion time was maintained constant. The plate was mounted on the press immediately after passing through the activator bath.

Several different etchants, both acidic and basic, were tried as activators with an applicator machine. In principle, any acid or base which effectively etched aluminum was usable. The bases were found to suffer from a decay in activity with time (**Figure 8**) because of the neutralizing action of carbonic acid originating in CO_2 absorbed from the air. On the other hand, direct plates treated with an acid activator were more sensitive to attack from the acidic moisture from the skin applied during handling. (In the case of phosphoric acid, such local attack produced aluminum phosphates which, although transparent, were still hydrophilic, so that scumming did not result.)

However, another major consideration was easy availability of etching solutions for the pressroom. For this reason, the highly basic commercially available developer solutions used for direct-imaging plates seemed especially attractive because of their wide availability. Such solutions are Mega developer, Itek Corp., Rochester, NY, and Onyx developer, 3M Corp., Minneapolis, MN.

Satisfactory results were obtained with such developers when they were diluted to give a pH of about 12.5 and the immersion time in the bath (defined as the time from the entrance of the leading edge of the plate into the bath to the time when it emerges from the bath) was about 5 s.

Another advantage of the direct-imaging developers is that they contain reagents which, although designed for reaction with silver halides, appear to extend the ability of the bath to serve as an effective activator even when the pH has fallen with time to relatively low values. (A study has shown that such constituents, e.g., hydroquinone, act in the same fashion as the nonetching promoters of ink uptake discussed earlier.) When such a commercial developer solution was used in a diffusion transfer machine, it was demonstrated that more than 70 plates in a 24-hour period could be treated without exhaustion of the bath (see Figure 8).

- *Press life*

A conventional offset-lithographic plate [1] typically shows wear on the press only after the production of tens of thousands of copies. If the backing material of the plate does not tear or buckle, the end of useful press life is usually caused by the wearing away of the thin film of cross-linked polymer which serves as the ink-attracting agent in the image region. The plate then gradually becomes blind; i.e., the ink concentration becomes inadequate in those areas where print is required.

In contrast to conventional plates, the wear of direct plates is not in the image areas, since such regions are represented by the thick polymer underlayer which will not wear away. On the other hand, the background area is associated with the presence of a very thin aluminum film which does wear away on the press, revealing the underlying hydrophobic polyester substrate. Unwanted ink therefore gradually begins to appear in the areas subjected to high wear; i.e., the background area gradually scums. Wear-induced scumming becomes detectable to the eye after several thousand copies for direct plates from DNP material fabricated according to the procedures previously described (Part II); such plates are therefore useful only as "short-run" plates. A study of the factors causing the undesired wear in the standard direct plate is presented in the following sections, together with a discussion of some techniques which permit wear reduction and hence greater press life.

Experimental techniques

Standardized testing procedures were adopted in order to obtain quantitative data related to wear of direct plates on the press.

For these press-life studies, either a large sheet of DNP material was used as a full-size plate, or alternatively a collection of small (5 × 5-cm) coupons was employed. In the former case, a test pattern (usually that of Figure 12 of Part II) was written on the direct plate with the IBM 4250 printer. In the latter case, a small test pattern of written rectangles was written on each coupon with a prototype of the IBM 4250 printer, and the coupons were then affixed with double-sided, pressure-sensitive adhesive tape to a conventional offset plate which served as a backing sheet. The composite plate was then mounted on the press. Such small coupons were convenient for small-scale experimental formulations of the DNP material, while the large plates were used in testing material on a larger scale (roll form).

As pointed out earlier, an activation treatment is needed before mounting a plate on the press. This treatment was carried out on full-size plates in the activator bath described above, but was awkward for the coupon samples. In the latter case the coupons were subjected to a plasma in air at reduced pressure for a few seconds, as mentioned earlier. The plasma treatment improved the hydrophilicity of the background area but did not improve ink uptake in the image area. This was not a serious disadvantage, since the *non*-image area was of predominant interest in press-life studies.

Since plate wear was manifested by a monotonic decrease in the optical reflectivity of the background area of successive copies, reflectometry measurements were employed to give a quantitative interpretation of this phenomenon.[14] A given direct plate could thereby be characterized by a curve of background-area reflectivity of the printed page vs. number of copies.

It was sometimes helpful to define a single-parameter description of this reflectivity curve corresponding to the end of useful life on the press. For this purpose several observers were shown samples of printed pages corresponding to different press run lengths (i.e., with scumming ranging from extremely light to moderate). The observers were asked to make a subjective comparison of these samples with unprinted paper in order to determine that sample for which scumming could barely be detected by eye. Upon examination with the reflection densitometer, it was found that such a visual scumming threshold corresponded to a decrease in reflectivity of about 0.8%. It should be noted that this value represents a very conservative estimate of scumming acceptability; it is thought that for many applications considerably more scumming, corresponding to appreciably longer press runs, would in fact achieve subjective acceptability. (B. J. Halter of IBM Boulder has noted that the visual scumming threshold also depends on the paper quality. For poor-quality paper, such as newsprint, a reflectivity decrease greater than 5% is acceptable.)

In some cases the *transmission* optical density of coupon samples of the direct plate was also monitored as a function of number of copies. For this purpose a coupon was detached from the backing sheet after a specified number of copies had been printed, and the ink was removed from the coupon by repeated washing with linseed oil, with a final rinse in ethanol. Light transmission

[14] Either of two instruments was used. The earlier experiments were carried out with a specially constructed instrument which utilized a white-light source which fed into a split fiber-optic bundle. One half-bundle was directed to the paper sample at near-normal incidence through a weak lens, illuminating an area about 5 mm in diameter; the reflected light was collected by a photodiode. The other half-bundle was collected by a reference photodiode. The signals from the sample photodiode and the reference photodiode were fed into a differential amplifier with logarithmic readout (Fiber-Optic Power Meter Model 450, United Detector Technology, Santa Monica, CA). The alternative reflectometer in later use was the "Chroma Meter II Reflectance" (Minolta Corp., Osaka, Japan) used in the white-light mode. An 8-mm-diameter area was measured with this instrument.

through an area about 5 mm in diameter was measured with a transmission optical densitometer.

Press life of standard short-run direct plate

Using the reflectometry technique, the background-area reflectivity change vs. number of copies was measured for a standard direct plate [**Figure 9(a)**]. The reflectivity change was normalized to zero for the first few hundred copies. This normalization was carried out because the first few copies exhibited not a decrease but a small *increase* in reflectivity compared with unprinted paper;[15] however, after about 100 copies the reflectivity reached a steady-state value, then slowly decreased as plate wear was manifested. The knee of the curve of Figure 9(a) corresponds to approximately 2000 copies; the visual scumming threshold discussed above corresponds roughly to 3000 copies.

In another experiment, after every few hundred copies the press was temporarily stopped and one of the set of direct-plate coupons was removed from the backing sheet for optical transmission measurements, as described above. It can be seen from **Figure 9(b)** that the transmission optical density began a gradual decrease very soon after the start of the press, with no sign of the knee observed in Figure 9(a). A clue to this difference in curve shape is furnished by examination of a corresponding set of optical micrographs of direct-plate coupons (**Figure 10**) made in a similar experiment. It is seen from Figure 10 that the aluminum was not worn away gradually and evenly over the entire surface, but rather that all the aluminum was worn away in selected localized areas; i.e., holes were formed. Such wear holes are seen to increase monotonically in concentration and size with increasing number of copies.

The discrepancy in the shapes of the curves of Figures 9(a) and 9(b) may be explained by noting that the reflectivity curve is determined not only by the wear of the aluminum, but also by the dynamics of the water–ink interaction on the press which influences the inking of the wear areas. As mentioned above, it was found in fact that if an aluminum-free area is less than about 10 μm in diameter, it will not accept ink. Scumming therefore increases very gradually at the beginning of the press run because such critically large wear areas are of only infrequent occurrence. However, for press-run lengths beyond the values corresponding to the knee, the wear areas have grown to the critical size where they can accept appreciable amounts of ink, and relatively rapid increases in scumming are observed.

Further microscopic examination showed that wear areas were initially associated with high peaks in the plate surface, as expected. Continued running on the press caused such wear areas to enlarge and scum, while elsewhere new small wear areas were created and subsequently enlarged and showed scumming. It should also be noted that material with pronounced fabrication-induced defects or material which scratched badly during electroerosion writing showed relatively short press life because wear areas associated with these defects were the first to grow to appreciable size and show scumming. For this reason fabrication-induced defects and scratching should be kept at a minimum (Parts I and II). For good-quality DNP material and good electroerosion writing conditions, initiation and growth of wear areas in the

[15] A small amount of lint picked up from the paper by the plate during press operation may cause the background reflectivity of the paper to increase with the number of printed copies. In normal operation this phenomenon usually required about 100 copies for a steady-state reflectivity to be reached.

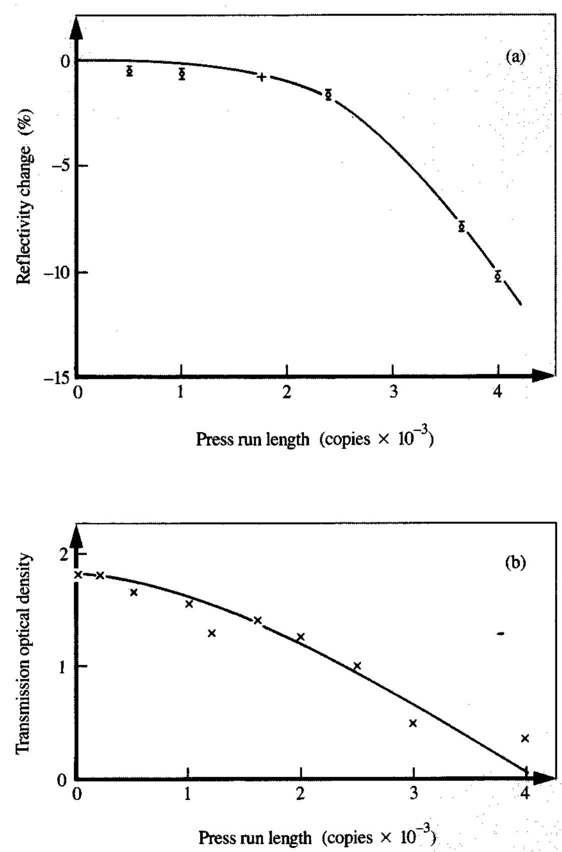


Figure 9

Dependence of optical characteristics on press run length, using a conventional-dampening press: (a) Reflectivity change of printed page vs. press run length for standard DNP material. Reflectivity was measured in background area. (b) Transmission optical density of plate vs. press run length for same standard DNP material used for (a). Transmission optical density was measured on coupons removed from press and cleaned after various press run lengths.

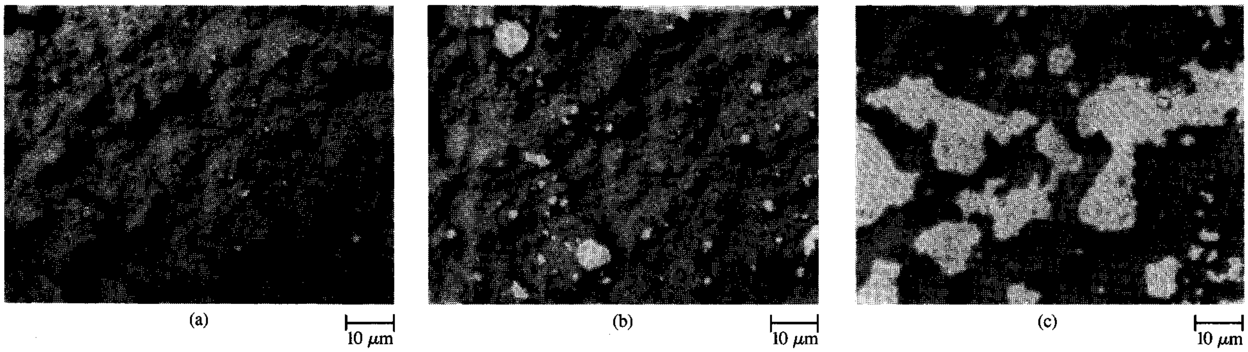


Figure 10

Transmission optical micrographs of standard direct plate after various press run lengths: (a) after 100 copies; (b) after 1000 copies; (c) after 3000 copies. White areas correspond to regions where aluminum was worn away. Micrographs were taken from different coupons which were removed from press and cleaned after various press run lengths. Conventional-dampening press.

aluminum occurred no faster in scratched areas than in virgin areas. The results described in this paper are typical for such virgin unscratched background areas.

The press-life data presented in this paper refer primarily to results obtained with the coupons discussed above. Results obtained with full-size direct plates were found to differ from corresponding results obtained with coupons, in that for the former samples plate wear was frequently emphasized in a band a few mm wide at the very top of the plate (leading edge) and also sometimes in areas corresponding to the suction cups used for paper feeding (see Section 2). These areas of preferential wear were outside the regions occupied by the individual coupons on their backing sheet, so that the press life for full-size plates was often substantially less than that corresponding to the coupons. Data obtained from coupons were nonetheless very useful in establishing trends and achieving detailed understanding.

Influence of press conditions on press life

Press life is determined by several factors, some associated with the structure of the direct plate and others associated with the operation of the press. Within the latter category, several observations gave indications that debris from the paper (paper "lint") is a major factor in determining press life:

1. For the integrated dampening system,[16] press wear of full-size direct plates was often first exhibited in the form of circles corresponding in position to the suction cups used for paper feeding. Some paper lint apparently adhered temporarily to the suction cups but was subsequently heavily redeposited in circles on the paper when the suction cups left the paper.
2. It was noted that the press life with bond paper was about a factor of 2 shorter than that with either electrophotographic copy paper or offset-lithographic paper. Pronounced paper lint pickup on the press is expected for bond paper.
3. In a special experiment, paper lint or simulated paper lint (powdered calcium carbonate) was sprinkled over a plate after every 100 copies. Clear signs of scumming and plate wear were seen at less than 1000 copies, which is considerably earlier than the 3500-copy life usually observed for a plate not so treated.

The abrasive nature of paper lint probably originates not in the soft cellulosic paper fibers, but in other constituents such as hard clay particles incorporated as fillers during paper manufacture. The presence of such particles is confirmed by **Figure 11**, which shows the paper lint stripped off the blanket from a background area with pressure-sensitive tape after a press run.

The following model is proposed to explain the mechanism of paper-lint-induced wear. It is postulated that during the working of the press a small amount of ink is absorbed ("emulsified") into the fountain solution, and that occasional tiny droplets of ink are therefore deposited on the blanket in the background area [5]. This phenomenon is observed with standard plates as well as direct plates, and is normally not directly responsible for scumming in either case. However, it is postulated that such ink droplets cause transfer of a small quantity of abrasive lint from the paper ("paper picking") to the

[16] See footnote 10.

surface of the blanket in the background area. As a result of the blanket–plate contact, this lint is able to cause wear of the direct plate in the background area.

The following special experiments with full-size standard direct plates were particularly instructive in confirming and elucidating this model:

1. A special printing regime was instituted on the integrated-dampening press, in which form-roller-to-plate contact was maintained but no paper was fed. After each 500th revolution of the plate cylinder, three sheets of paper were fed through the press in order to monitor the quality of the printed page. For this procedure no background scumming was found after more than 18 000 copies; as seen from **Figure 12**, the measured reflectivity in fact *increased* during this interval.
2. A special printing regime was instituted on the integrated-dampening press in which the press was operated normally except that the blanket was cleaned every 50 copies. The press life in this case was greater than 8000 copies (Figure 12).
3. A special printing regime was instituted on the conventional-dampening press in which paper was fed through normally and the water form-roller contacted the plate normally, but the ink form-roller was not in contact with the plate. However, at every 500th revolution of the plate cylinder, two sheets of paper were fed through the press with the ink form-roller in its normal inking position. No background-area scumming was found in this case after more than 20 000 copies (Figure 12).
4. A special printing regime was instituted on the integrated-dampening press in which a sheet of polyester film was installed and run as a plate for a few copies after each set of 50 copies was printed from a direct plate containing a standard test pattern. The polyester sheet covered only half of the area normally occupied by the direct plate; the other half was left blank. In this fashion a relatively heavy ink deposit was given to one side of the blanket after every 50 copies of the direct-plate press run. Under these conditions it was found that the wear of the plate on the additionally inked side (the side corresponding to the polyester sheet) was appreciably higher than on the untreated side; after 1000 copies the transmission optical density of the (cleaned) plate on the additionally inked side was 0.1–0.15 less than that of the noninked side, while the corresponding difference in reflectivity of the printed paper was about 0.5%.
5. It was noted that the press life obtained with the integrated-dampening press gradually decreased as the blanket was used over a long period of time. Heavy, unremovable deposits of organic matter were observed on the surface of this old blanket toward the end of its useful life. However, upon installation of a fresh blanket the press life increased from about 1000 to 3000 copies.

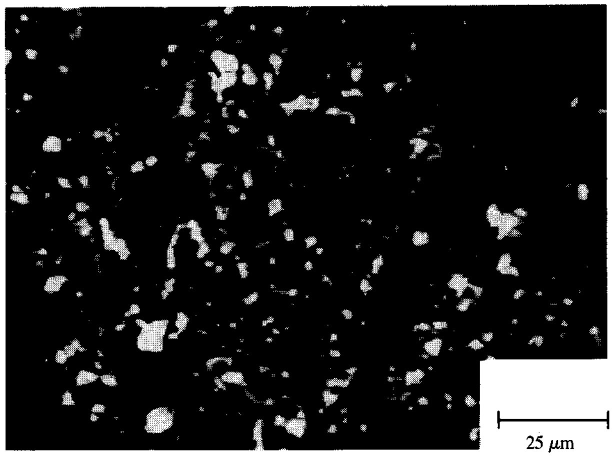


Figure 11

Scanning electron micrograph of paper lint stripped from blanket after press run of standard direct plate. Integrated-dampening press.

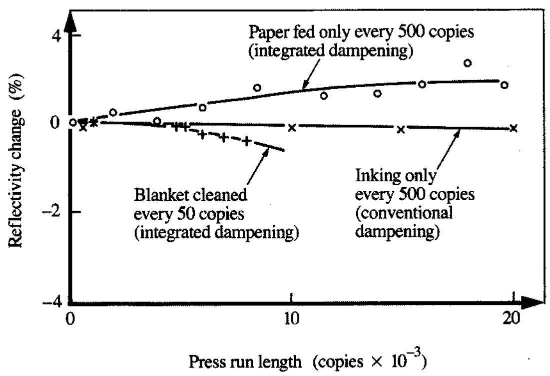


Figure 12

Reflectivity change vs. press run length for special printing regimes of standard direct plates. Reflectivity was measured in background area of printed paper. 1) Form-roller-to-plate contact was maintained, but paper was fed only after each 500th revolution of plate cylinder, at which time three test sheets of paper were fed. Integrated-dampening press. 2) Blanket was cleaned after every 50 copies. Integrated-dampening press. 3) Ink form-roller was in contact with plate only after every 500 plate-cylinder revolutions, at which time two test sheets of paper were printed with ink form-roller contact. Conventional-dampening press.

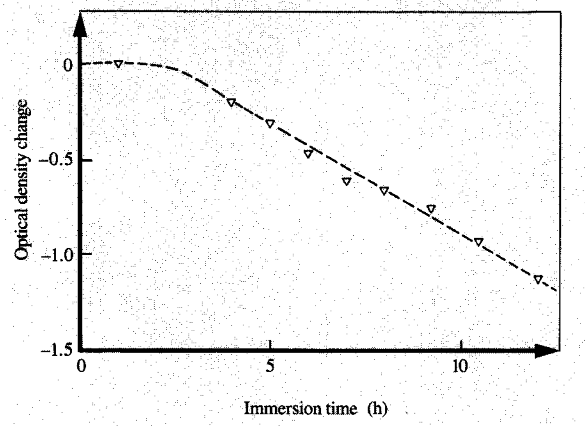


Figure 13

Optical density change vs. immersion time in fountain solution. Standard direct-plate coupons were placed in stirred fountain solution. Fountain solution used was diluted as used on the press; pH was about 4.

6. The press life was found to be dependent on the printing speed. Thus, decreasing the speed of the integrated-dampening press from 10 000 to 3500 copies per hour increased press life by about 2000 copies.
7. The press life was found to increase with decreasing ink tack. It was found, for example, that with a conventional-dampening press the press life decreased from about 6000 copies with a medium-tack ink[17] to about 1000 copies with a high-tack ink.[18]

The results of Experiment 1 reconfirm the importance of paper lint in the wear process. The increased press life with frequent blanket cleaning (Experiment 2) shows that the accumulation of the lint on the blanket is instrumental in causing the wear. The general role of ink in promoting pickup of lint from the paper is shown in Experiment 3, while Experiments 4 and 5 show that the introduction of ink to the blanket surface promotes paper-lint pickup and consequent plate wear. It is known [1] that increased press speed and increased ink tack exacerbate paper picking (Experiments 6 and 7).

Given that ink emulsification promotes paper picking, which in turn leads to plate wear, certain precautions may be taken to maximize press life. Relatively low-tack ink and pick-resistant paper should be used in conjunction with an unworn blanket at relatively low press speeds to reduce picking. Ink emulsification should be minimized by using the lowest practical flow rate of fountain solution, and by using an ink which is as water-rejecting as possible. (The water-rejection property of an ink may be increased by addition of a component such as No. 30 Aqua Varnish, Van Son Holland Ink Corp. of America, Mineola, NY.) When these measures were instituted, press life was extended from about 2500 to 4000 copies on the integrated-dampening press.

It may also be noted that if the ink itself is abrasive, reduced press life results. In this case the wear mechanism would involve emulsification and transfer of the ink to the background area, as usual. However, the mechanism of subsequent incorporation into this ink of abrasive lint by paper picking need not be invoked to explain the resulting plate wear, since the ink already contains abrasive agents. This effect was noted for magnetic ink[19] containing iron oxide particles, which are hard compared to conventional carbon-black particles. Magnetic inks typically give a press life of 1000–2000 copies less than that obtained with standard inks.

Another major factor in determining press life is the activation treatment. If the plate is exposed to the activator bath for too long a period, tiny voids in the aluminum film may be formed which grow as a result of wear on the press. Care must therefore be taken to control the activation time and pH of the activator bath so that etching is stopped when the background area is cleaned and excess residual aluminum in the written area is removed (Section 2). In this connection it is noted that when the standard overlayer (Part II) was replaced by an experimental overlayer having a slightly acidic nature,[20] an increase in press life of about 1000 copies was observed, presumably because of partial neutralization of the basic activator bath in the background area.

Another potentially important factor in determining press life is the pH of the fountain solution used on the press. Standard fountain solutions have a pH of about 4, which is acidic enough to etch aluminum slowly. The etching action was studied by placing a coupon of plate material into a container of standard fountain solution[21] under stirring action. The coupon was periodically removed and its transmission optical density was measured. The results of this experiment are shown in **Figure 13**, where the change in optical density is plotted against immersion time. It is seen that there is little loss of optical density in the first hour, but that the loss subsequently increases rapidly. For a standard duplicator press running at 7000–10 000 copies per hour, it would therefore seem that the mechanical wear mechanisms

[17] Van Son 40904 Black, Van Son Holland Ink Corp. of America, New York, NY.
[18] Omni Black 80800, Gans Ink and Supply Co., New York, NY.
[19] Van Son 68841 Black, Van Son Holland Ink Corp. of America, Mineola, NY. Such inks are used, e.g., in the printing of bank checks.
[20] Polyvinylmethyl ether maleic anhydride, Gantrez AN139, GAF Corp., New York, NY.
[21] All Purpose Fountain Solution, Van Son Holland Ink Corp. of America, Mineola, NY. A 7:1 dilution with water was used.

discussed above would cause scumming before the etching mechanism could become decisive. For this reason, when the standard acidic fountain solution was replaced by a *neutral* fountain solution,[22] no significant increase in press life was observed.

Influence of DNP structure on press life

Assuming that the press is operated under conditions which give a reasonable press life, the question of how the structure of the DNP material affects the press life must next be examined.

It is postulated that in an underlayer of inadequate hardness, the high peaks of the rough underlayer are subjected to appreciable flexure during press operation, thus causing accelerated local wear of the aluminum film. The hardness of the underlayer is affected by the silica concentration, the strength of the bond of the silica particles to the binder matrix, the cross-linker concentration, and the cure treatment (Part II).

Direct measurements showed the anticipated increase of microhardness with increased silica PVC (Part II). Correspondingly, studies on the press showed that an increase of the silica PVC from 10% to about 20% caused an increase in press life from several hundred to several thousand copies. The influence of cross-linker concentration on press life is illustrated in **Figure 14**. In addition, it may be noted that an increase of underlayer thickness from about 3.8 μm to 6.2 μm led to a press-life increase from 2000 to about 4000 copies, also probably because of the consequent decrease in flexure on the press.

Another parameter having an important effect on press life is the aluminum thickness, as shown in **Figure 15**. The press life was found to increase with increasing aluminum thickness, perhaps because the occurrence of tiny pinholes was thereby suppressed. This would lead to improvement in wear resistance because, as pointed out previously, such pinholes act as nucleating centers for press wear.[23] However, as seen from Figure 15, an increase in aluminum thicknesses substantially above the usual value (optical density ~2) was required to give an appreciable increase in press life.

Despite the demonstrated improvements in press life given by effective increases in underlayer hardness or in aluminum thickness, the discussions presented previously (Part II) show that considerations of the effects on other properties make it difficult to change the values of these parameters. A search for more practical techniques for press-life improvement achieved some success when it was realized that there was a close relationship between press life and adhesion of the aluminum to the underlayer. This

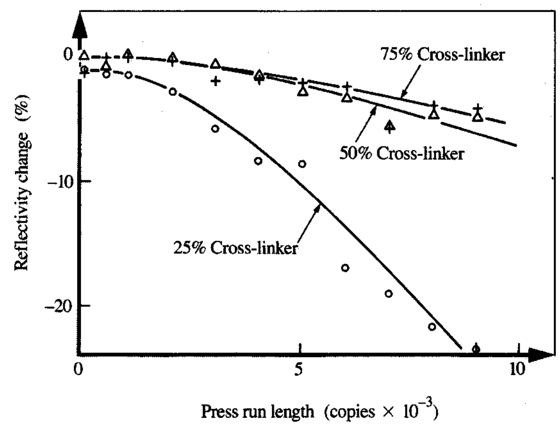


Figure 14

Reflectivity change vs. press run length for direct-plate coupons having underlayers with various concentrations of isocyanate cross-linker. Reflectivity was measured in background area of printed paper. Conventional-dampening press.

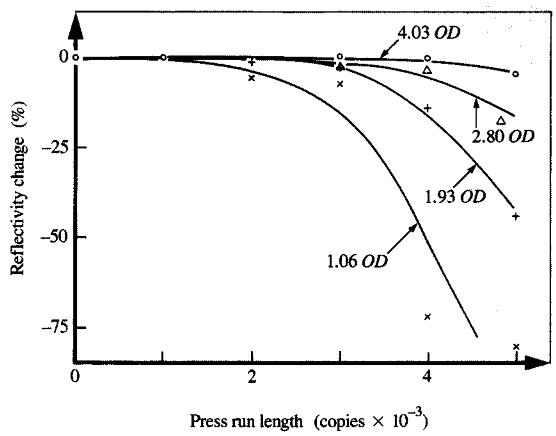


Figure 15

Reflectivity change vs. press run length for various values of the transmission optical density of direct-plate coupons, i.e., corresponding to various aluminum thicknesses. Reflectivity was measured in background area of printed paper. Conventional-dampening press.

[22] Adam N Fountain Solution, Graphic Arts Technical and Consulting Services, Long Island City, NY.

[23] In this connection it may also be mentioned that an enhancement of press life of several thousands of copies was observed if activation was performed not with the usual solutions but with either sodium silicate solution or solutions containing phosphoric acid and phosphates. It is thought that the aluminum silicates or aluminum phosphates thereby formed on the aluminum film may act to increase the effective thickness of the composite film, and thereby also to close tiny pinholes.

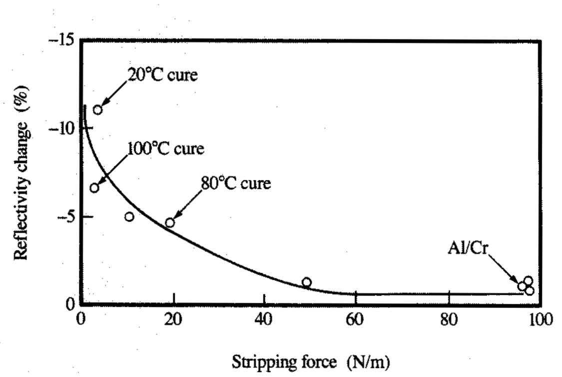


Figure 16

Reflectivity change after 10 000 copies on the press vs. force required to strip aluminum from underlayer. Samples were 5 × 5-cm coupons made by various techniques. Reflectivity was measured in background area of printed paper. Integrated-dampening press.

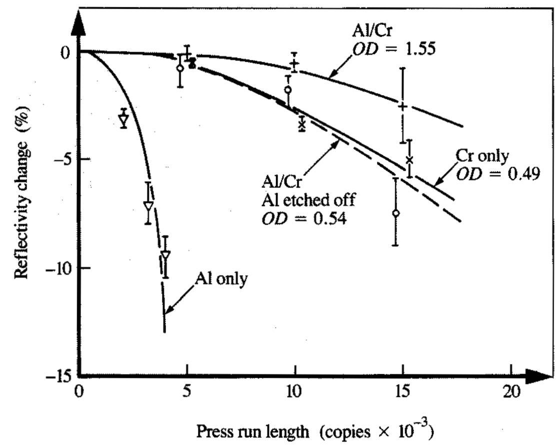


Figure 17

Reflectivity change vs. press run length for coupons made with various metal films: 1) Standard aluminum film. 2) Aluminum film with chromium underfilm (total transmission optical density 1.55). 3) Only chromium film deposited (transmission optical density 0.49). 4) Original aluminum film with chromium underfilm (total transmission optical density 1.55), then aluminum etched off, leaving only chromium underfilm with transmission optical density 0.54. Reflectivity was measured in background area of printed paper. Conventional-dampening press.

relationship was demonstrated by "peel" tests in which the aluminum–substrate adhesion was measured directly. The results of such tests are shown in **Figure 16**, which shows a plot of background-area reflectivity change of the paper after 10 000 copies as a function of adhesion as measured by the stripping force.[24] The points on the curve represent 5 × 5-cm samples made by various techniques, some of which are subsequently discussed. Those samples which had better aluminum adhesion to the underlayer clearly demonstrated less scumming, presumably because wear on the press involves breaking the aluminum–underlayer bond.

Methods of improving aluminum adhesion

The fundamental mechanisms of metal–polymer adhesion are still under discussion [6]; nevertheless, it is known that several techniques permit improvement of this adhesion.[25] Of the adhesion-enhancement methods explored, those showing most promise for practical application included 1) exposure to plasma before aluminization, 2) incorporation of a thin adhesion-promoting underfilm between the aluminum and the underlayer, and 3) modification of the underlayer composition.

Curing treatment Heating is known to reduce the concentration of surface contaminants such as oil, water, or other loosely bound material at a polymer surface [7], so that subsequently deposited metal can bond directly to the substrate. Effects of cure treatments of increasing temperature can be seen in Figure 16. In other experiments the radiant energy of the evaporation source in the metallizer was itself used to preheat a moving web before it passed to the zone of highest deposition rate. In special experiments the underlayer temperature was further increased by reducing the web speed and by incorporating carbon black into the underlayer in order to increase the radiant energy absorption. The adoption of these measures led to an increase in press life of about 1000 copies.

Exposure to plasma Another treatment which has been used to improve the adhesion to polymers involves their exposure to a gas plasma at reduced pressure [8]. To apply this treatment in the present application, small coupons of DNP material were subjected to a plasma in air just prior to aluminization, using reduced pressure in a laboratory apparatus designed for plasma ashing (LFE Corp., Clinton, MA). The results demonstrated a consequent increase in

[24] For the stripping-force measurements a 3-cm × 4-cm piece of polyester film about 75 μm thick was epoxy-bonded to the aluminum side of a 3 × 3-cm piece of DNP sheet, so that at least 5 mm of unbonded polyester film was left at an edge. A strip 5 mm wide was then cut from this composite in such a fashion that the resulting sandwich retained an unbonded tab of polyester. The back (polyester substrate) side of the DNP portion of the sandwich was then epoxy-bonded to a glass microscope slide, and the entire structure was cured at 100°C for 8–10 h. The glass slide was then fastened to the bed, while the tab was clamped into the movable jaws, of a tensile-strength measurement instrument (Instron Engineering Corp., Canton, MA), and the stripping force was measured.

[25] Because of the greater energy of the incoming metal atoms, some improvement of adhesion is often found for sputter-deposited films compared to films deposited by thermal evaporation. However, in the present studies no difference in adhesion or in consequent press life was observed between DNP samples prepared in these two different ways.

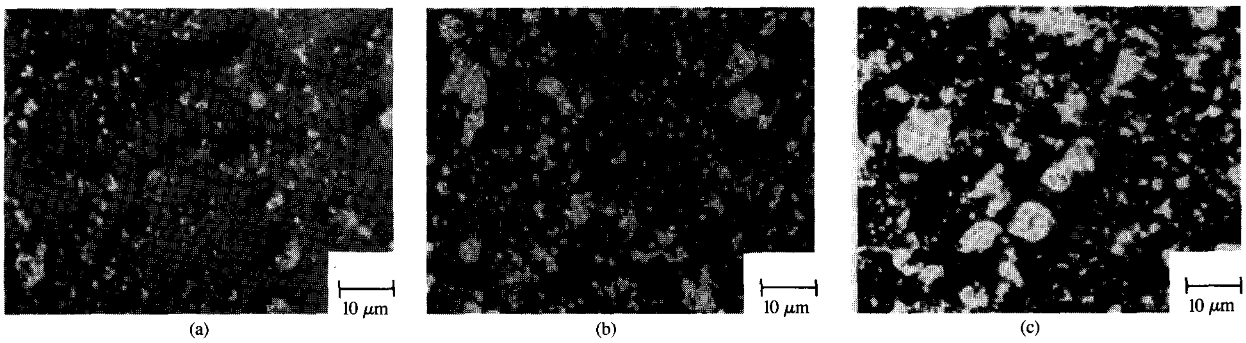


Figure 18

Optical micrographs of direct plate having chromium underfilm after various press run lengths: (a) after 10 000 copies; (b) after 25 000 copies; (c) after 40 000 copies. White areas correspond to regions where aluminum was worn away. Micrographs were taken from different coupons which were removed from press and cleaned after various press run lengths. Conventional-dampening press.

press life to about 11 000 copies. The effectiveness of plasma treatment for adhesion enhancement may be explained alternatively by microroughening of the surface, by increasing the chemical reactivity of the surface, or by an etching action which serves to clean weakly bonded material from the surface. It is not clear whether or not activated oxygen plays a major role in this process; cellulosic material should already be well oxygenated [9].

The plasma technique could be carried out on a larger scale by passing the web of a master roll of DNP material through an appropriate plasma chamber just before aluminization.[26] Tests with coupon samples have shown that the effectiveness of the plasma pretreatment is maintained for several days, so that a reasonable time could elapse between the plasma treatment and the aluminization.

Incorporation of underfilm It is well known in the field of vacuum metallization that thin underfilms of such materials as nickel, titanium, or chromium enhance the adhesion of metallic films to nonmetallic substrates [10]. The consequent anticipated increase in press life of DNP material incorporating such underfilms was confirmed by experiment. Since initial studies showed that samples with underfilms of chromium had a longer press life than samples with underfilms of nickel or titanium, further investigations were made of underfilms consisting of either metallic chromium or chromium compounds. (Although some studies were carried out with chromium films prepared by vacuum evaporation, these films were deposited primarily by sputtering [10]; the overlying aluminum film was deposited either by sputtering or evaporation without noticeable change in DNP properties.)

Al/Cr: Behavior on press The press-life improvement given by an underfilm of chromium is illustrated in **Figure 17**, which shows the optical reflectivity change vs. number of copies for typical standard and chromium-underfilm direct plates. (The thickness of a chromium underfilm was measured alternatively by its optical density or its mass/unit area as determined by X-ray microprobe or quartz crystal monitor, or by direct measurement by a profilometry technique. The measurement was usually converted to thickness in nm.)

Transmission micrographs of aluminum-on-chromium coupons taken after various numbers of press copies (**Figure 18**) show hole formation and growth effects similar to those presented in Figure 10 for an aluminum-only plate. The major difference between the two cases is primarily the very much slower hole formation in the aluminum-on-chromium case, as is also demonstrated by transmission densitometry (**Figure 19**).

Besides enhancement of adhesion, a secondary cause of the observed increase in press life with chromium underfilms may be that the chromium sometimes remained after the aluminum was worn away (separation of the aluminum from the chromium underfilm[27]), and that it is the hydrophilicity of this remaining chromium which prevented the undesired scumming. (It may be noted that chromium metal is often used for the *background* surface for conventional long-run bimetallic plates [1].) The possibility of the latter mechanism is shown by the curves of Figure 17 corresponding to chromium films without aluminum; in one case the overlying aluminum was etched

[26] A *corona* treatment (electric discharges in air at atmospheric pressure) is a standard adhesion-enhancement treatment given to polymeric webs before coating with polymer films. When such a corona treatment was used before metallizing DNP material, no significant increase in press life was observed. This treatment is apparently not as effective as the plasma treament.

[27] It may also be noted that peel tests sometimes showed a break in adhesion at the aluminum–chromium interface.

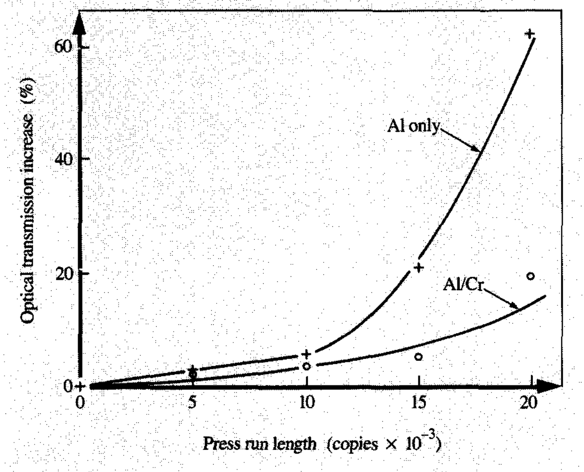


Figure 19

Change in optical transmission vs. press run length for DNP material corresponding to 1) Standard aluminum-only plate. 2) Direct plate with a chromium underfilm; the same DNP material was used for obtaining the micrographs of Figure 9. Conventional-dampening press.

off, while in the other case no aluminum was ever deposited. In both cases the thin chromium-only coupons showed an appreciable increase in press life over that of the aluminum-only coupon,[28] but coupons with aluminum on chromium had the longest press lives of all.

In further studies of the latter issue, analysis by X-ray microprobe was used to examine the ratio of aluminum to chromium for coupons which had been on the press for many thousands of copies. This technique showed that both the aluminum and chromium layers were intact in some regions, while they wore away together in other regions which consequently became receptive to ink. However, in still other regions the data indicated that a thin chromium film remained behind after wear of the aluminum. This question of the aluminum-to-chromium ratio in the wear areas was also examined by optical microscopy, which showed wear holes after many thousands of copies [**Figure 20(a)**]. The aluminum overfilm was then etched off the sample, again showing by the subsequent elimination of the optical contrast associated with some of the holes [**Figure 20(b)**] that in some cases a residual film of chromium remained in the wear holes, while in other cases the chromium had also been worn away.

[28] A coupon with pure chromium metal film without aluminum showed a very long press life (>75 000 copies without any sign of deterioration) for a chromium thickness of about 45 nm. However, such DNP material is impractical because of the very poor writing quality achievable with chromium due to its high melting point (see below).

The visual scumming threshold was found for chromium underfilms of various thickness by taking, for each underfilm thickness, a curve of reflectivity change of the printed paper vs. number of copies. In this way a curve of scumming threshold vs. chromium underfilm thickness was generated (**Figure 21**).

Some insight into the shape of the curve of Figure 21 may be obtained from other studies involving chromium on various rough substrates, which showed that only partial coverage of the surface by the chromium occurred until average thicknesses between 5 and 15 nm were reached [11]. It therefore seems reasonable to assume that the

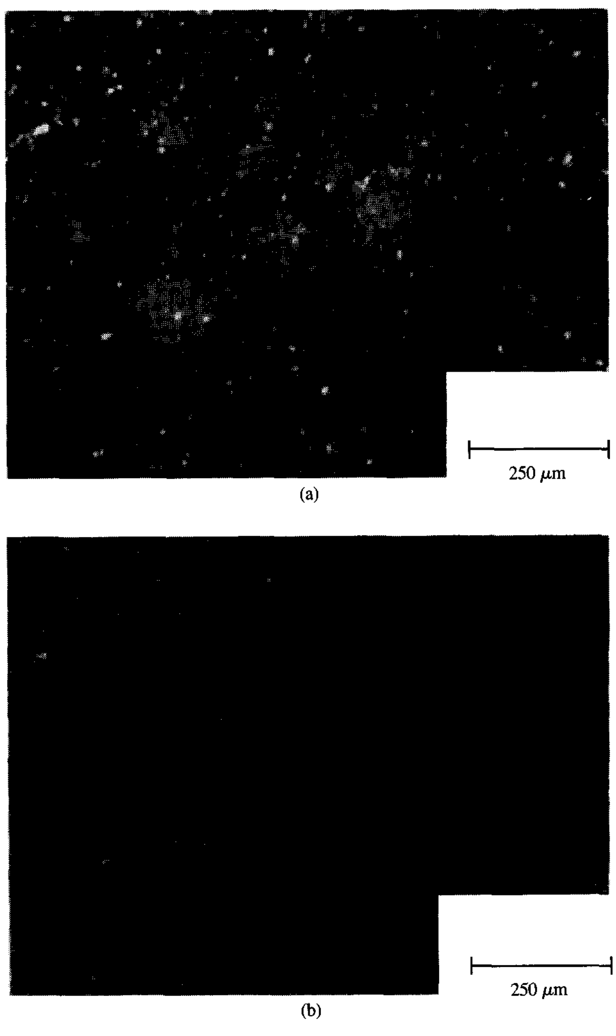


Figure 20

Micrographs of direct-plate coupon with chromium underfilm (2.3 nm thick) after 80 000 copies on press: (a) after removal of coupon from press and cleaning; (b) after etching in sodium hydroxide solution to remove aluminum. White areas which appear in both (a) and (b) indicate wear holes through aluminum *and* chromium, while white areas appearing in (a) but not (b) indicate wear through aluminum but not chromium film.

improvement of press life with increasing chromium underfilm thickness originates in the corresponding increase in surface coverage of the chromium. This mechanism may also explain the tendency of the curve to saturate for higher values of chromium thickness.

An important difficulty with chromium-underfilm plates is the poor reproducibility of press-life results, particularly for underfilms about 5 nm and thinner. Other thickness series showed curves considerably different from that of Figure 21; e.g., for a 5-nm underfilm, press-life values ranged from about 10 000–50 000 copies among these series. This irreproducibility presented a serious problem since, as pointed out below, the maximum usable chromium underfilm thickness is no more than 3–5 nm in practice.

Al/Cr: Influence on writing quality It is clear that the electroerosion writing process requires more energy to remove chromium (melting point 1890°C) than aluminum (melting point 660°C). The quality of the electroerosion writing is thus expected to worsen as the thickness of the chromium underfilm increases. This expectation is borne out by experiment (**Figure 22**), which shows a decline in writing efficiency for underfilm thicknesses substantially above 3 nm.

The cause of this phenomenon appears to lie in the difficulty of removing metal at the stylus contact during phase 1 of the applied pulse (Part I). Detailed study showed that the amplitude and length of the phase 1 pulse were very large for the aluminum-on-chromium compared with the aluminum-only case, indicating inadequate contact-breaking by the phase 1 pulse in the former case; the prolongation of the phase 1 pulse in turn led to inadequate writing.[29] To ensure good writing quality, the chromium underfilm should thus be as thin as possible commensurate with an appreciable increase in press life. A thickness from 3 to 5 nm appears to be practical. However, as pointed out earlier, under these conditions shorter press life and less reproducible results were found than would be expected for thicker underfilms.

Al/Cr cermets In order to obtain reproducibly longer press life, investigations were made into the metal–insulator films known as *cermets* [10]. The motivation for investigating such films was to increase the underfilm resistivity above that for chromium metal, so that phase 1 could be completed in a shorter time. In this way it was hoped that an underfilm could be deposited having a thickness beyond that which was practical for

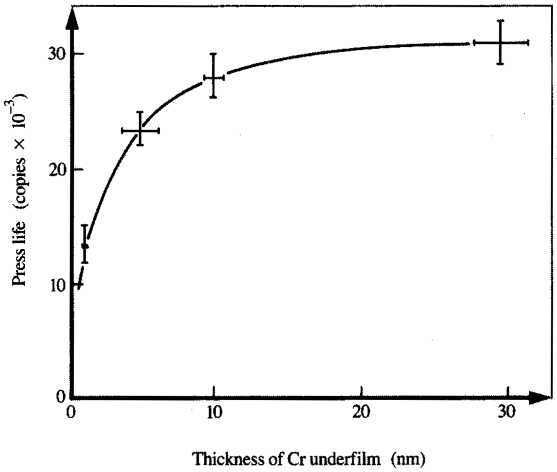


Figure 21

Press life (based on visual scumming threshold) of direct-plate coupons with chromium underfilm vs. thickness of underfilm.

chromium-metal underfilms having good writing quality, thereby achieving a prolonged, reproducible press life.

In this scheme, chromium–cermet films would be made by codeposition of chromium with another element. For example, chromium–silicon films have been made in this fashion [12, 13], thereby giving films of enhanced resistivity. Appropriate sputtering targets were not readily available in this work, however, so that attempts were made to achieve the increased resistivity by *reactive* sputtering, where during the sputtering operation a gas was present which reacted with the deposited metal. The primary gas employed for emission of chromium from the target was argon; additions of either oxygen or nitrogen were made to the argon to act as the reactive gases.

It was found that the properties of the underfilm varied with the partial pressure of the nitrogen present. (Studies have shown that in addition to the nitride, a high proportion of oxide is incorporated into chromium films reactively sputtered with nitrogen and argon gases. The origin of the oxygen is not clear; it may originate in the walls of the sputtering chamber.) An overall summary of several experiments is given in **Table 4**; all plates referred to in this summary consisted of coupons metallized in a laboratory magnetron-sputtering chamber. It is seen that sputtering in pure argon gave chromium-metal underfilms with low resistivity, poor writing quality for thicknesses greater than 3–5 nm, and moderate but irreproducible press life for thicknesses corresponding to good writing quality.

[29] It is known (Part I) that a rougher substrate results in a desirable suppression of phase 1; experiments reconfirmed this result for chromium-underfilm DNP material. For a given chromium-underfilm thickness, the underlayer roughness should thus be large. However, the underlayer roughness is fixed by the considerations given previously (Part II).

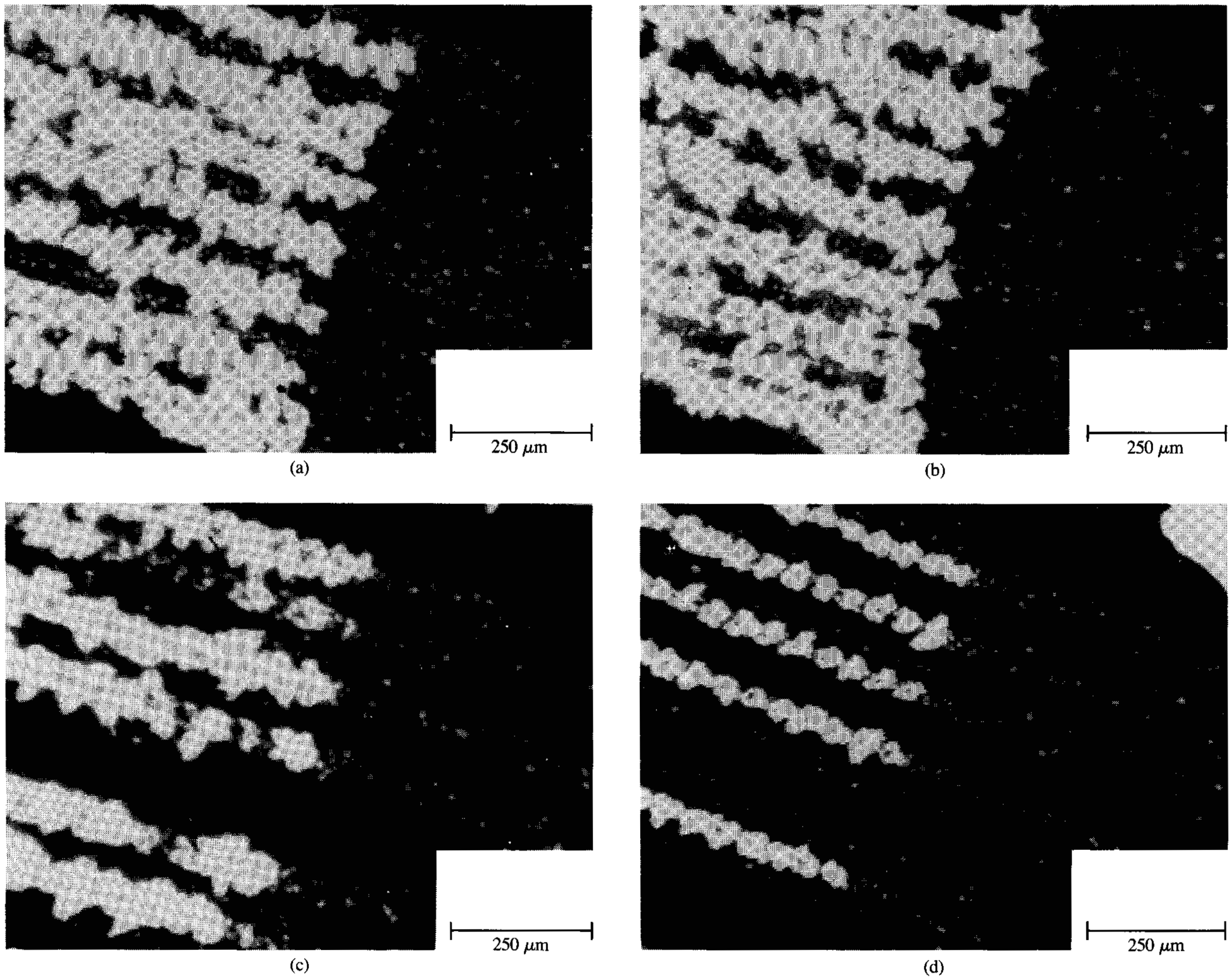


Figure 22

Optical micrographs of written area of direct-plate coupons having various thicknesses of chromium underfilm: (a) no chromium; (b) 2 nm chromium; (c) 3 nm chromium; (d) 10 nm chromium. Written with prototype of IBM 4250 printer.

Table 4 Chromium–cermet underfilm characteristics.

Sputter gas	*Resistivity* ($\mu\Omega$/m)	*Writing quality*	*Press life* (copies $\times 10^{-3}$)
100% A	Low: ~1	Poor: >3–5 nm	<30, varies
90% A, 10% N_2	Medium: ~3–6	Poor: >25 nm	50–90
58% A, 42% O_2	Medium: ~9	Poor: >25 nm	15–50
100% N_2 or 90% N_2, 10% A	High: ~100–1000	Poor: >10 nm	25–35

Press life given for underfilms in the thickness range showing good writing quality.

The addition of 10% nitrogen to the argon increased the resistivity by a factor of 2–5 so that the films could be classified as cermets, resulting in good writing quality for thicknesses up to 25 nm; the press life was far higher for such films, falling in the range of 50 000–90 000 copies. As also shown in Table 4, the cermets made when oxygen was added instead of nitrogen showed good writing quality at intermediate resistivity values, but the press life was not reproducibly high. Table 4 also shows that when a *high* concentration of nitrogen was added to the argon, the resulting films had very high resistivity but showed good writing for thicknesses only up to 10 nm, with consequent poor press life.

Studies with the optical microscope, using the etching technique described above, have suggested that both the aluminum and the underfilm were removed during the writing operation. The fact that the writing quality was poor for thicker underfilms of high resistivity (Table 4) may therefore be explained by postulating that such films are so ceramic-like that they resist the electroerosion process; i.e., underfilm disintegration could not be achieved during writing. Resistivities of 100 $\mu\Omega$/m thus appear to be too high (underfilm disintegration limitation) for good writing quality, while 1 $\mu\Omega$/m is too low (phase 1 limitation). Values of roughly 5 $\mu\Omega$/m seem to be optimal.

These data thus indicate that the best properties are obtained with a sputtering gas of 10% nitrogen and 90% argon. Results for a typical series of underfilms of various thicknesses made under these conditions are shown in **Table 5**. Here it is seen that good writing quality and prolonged, reproducible press life were found for a wide range of underfilm thicknesses and considerable variation in overfilm (aluminum) thickness.

Limited scaled-up coating of chromium–cermet underfilms on master rolls was carried out using a roll-coating sputtering system operating under reactive sputtering conditions.[30] Despite the fact that the resulting chromium–cermet compositions were far from optimal, press-life values of more than 10 000 copies were nevertheless achieved.

4. Conclusions

The reproduction of scratch and pinhole defects in the direct negative as well as direct exposure through the aluminum can be minimized by careful attention to exposure control.

A simple, practical method for prepress activation of the direct plate was introduced in order to avoid initial scumming and blinding problems. This technique is semiautomatic, and involves solutions and apparatus readily available to the pressman.

The primary press-life-limiting mechanism was identified as wear of the direct plate originating in paper-lint pickup. Some extension of press life is possible by adjustment of the press conditions. Longer press life was also obtained by certain structural modifications of the DNP, the most promising of which involved measures which improved the aluminum–underlayer adhesion. These measures included the following:

1. Exposure of the underlayer to a plasma before aluminization.
2. Incorporation of a thin "underfilm" containing chromium or chromium compounds between the aluminum film and the underlayer.
3. Changing the composition of the underlayer binder from CAB to an alternative formulation, e.g., polyvinyl butyral.

The largest, most reproducible values of press life were obtained with underfilms of chromium compounds.

[30] Test coatings were applied at Martin Processing Inc., Martinsville, VA.

Table 5 Chromium–cermet underfilm thickness series.

CrX underfilm			*Aluminum optical density*	*Press life* (copies × 10^{-3})
Thickness (nm)	*Optical density*	*Resistivity* ($\mu\Omega$/m)		
5	0.28	5.5	1.23	49 ± 5
10	0.42	4.6	1.74	53 ± 5
20	0.65	5.6	1.20	39 ± 3
25	0.85	4.1	1.23	45 ± 5

1. Used 10% nitrogen, 90% argon during sputtering.
2. Writing quality was good in all cases.
3. 25-nm coupon was made with 5% nitrogen, 95% argon.

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