The influence of electroless plated chromium on printing properties of aluminum offset printing plate

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A method for the improvement of the printing properties of offset printing forms is presented. Specimens of technical aluminum were electrochemically roughened and treated in different alkaline solutions of chromium chloride in order to chemically deposit a chromium layer. The composition of the surface layer was investigated by EDAX. Chromium was found on the specimens that had been treated in an alkaline solution of chromium chloride, while no chromium was found on chemically untreated specimens or on specimens that had been treated in an alkaline solution without chromium chloride. The spectral reflectance from treated and non-treated specimens was also measured. The chromium-treated specimens were brighter than the non-chromium-treated ones. The wettability of the chromium-treated samples was compared with the wettability of the non-chromium treated samples by measuring the contact angle with water and the wetted area. The chromium-treated samples showed increased wettability compared with the non-chromium-treated samples. A printing test was performed under real printing conditions. Control prints was analyzed densitometrically and statistically. The chromium-treated printing forms gave clearer prints than the non-chromium-treated printing forms under all printing conditions.

Keywords: aluminum, chromium, electroless deposition, printing plates.

INTRODUCTION

A difference in hydrophilicity between the printing (hydrophobic) and non-printing (hydrophilic) elements of an offset printing form is essential for the printing process. During the printing process, a dampening solution is first applied onto the printing form generating a thin water layer on the non-printing elements. The dampening solution consists mostrly of water (min 80 wt. %). It could also contain acid, buffer, gum arabic, surface-active substances (*e.g.,* 2-propanol) and other substances, which increase the stability of the dampening solution and the whole printing process. In the second step, ink is applied on the printing form. Normally ink does not cover the elements wetted by a dampening solution, which serves as an ink repellent. The non-printing elements are the parts of the mechanically or electrochemically roughened aluminum base which are not covered with a polymer layer.[1]


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The stability of the non-printing elements is one of the most important requirements for obtaining high circulation with good quality. A possible solution for increasing the stability of the non-printing elements is the anodic oxidation of the aluminum plate, whereby it is possible to form a thick anodic oxide layer, with high wear resistance and greater hydrophilicity than a spontaneously formed oxide layer. However, such a method requires special equipment and consumes a lot of electric power.[2]

Another method for increasing the non-printing element stability is to electroplate chromium onto other metals. A chromium layer on bimetallic printing plates shows excellent wear resistance and such plates are especially suitable for the highest circulation (up to a million) and the highest printing quality, but they are even more expensive than anodized plates. The chromium layer is formed electrochemically, and so a lot of electrical power is consumed.[3]

In addition to plating by electrodeposition, spontaneous deposition of a metal is also known. This occurs when a metal is immersed into a solution that contains ions of an electrochemically more noble metal. The phenomenon that a more noble metal is deposited from its solution onto a less noble metal is called cementation.

In this work, a chromium layer was deposited by cementation (*i.e.*, electroless), directly onto aluminum, during the development of an offset printing plate.[4] The method is quite simple, and the chromium layer could improve the properties of the non-anodized aluminum offset printing plates.

The overall cementation reaction is shown in Eq. (1).

$$(k/m)M_1(s) + M_2^{k+}(aq) = (k/m)M_1^{m+}(aq) + M_2(s) \tag{1}$$
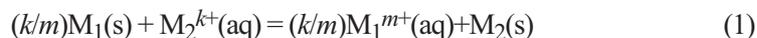
where M_1 is the less noble metal and M_2 is the more noble metal.[5]

The cementation reactions of copper (reaction potential $Cu^{2+} + 2e^- = Cu$: 0.337 V)[6] and zinc ($Zn^{2+} + 2e^- = Zn$: –0.763 V) onto aluminum ($Al^{3+} + 3e^- = Al$: –1.662 V) from alkaline solutions are well known.

The electrode potential of aluminum is more negative than that of most other metals. Based on this fact, it could be assumed that aluminum could reduce and deposit most other metals. This is not an advantage when plating aluminum in usual electrolytes, so the aluminum was prepared for plating by zincate or stanate (Alstan) treatment. To the best of our knowledge until now there are no data concerning the cementation of chromium ($Cr^{3+} + 3e^- = Cr$: – 0.744 V). In this work, an attempt was made to deposit chromium layers from alkaline solution on previously roughened aluminum at room temperature. The main idea was to obtain in a simple way something like a bimetallic plate. Then, the influence of the chromium layer on the surface properties of aluminum was considered.

EXPERIMENTAL

In this work, previously roughened aluminum specimens were chromium-plated by cementation. The specimens of technical aluminum (Al 99.5 wt. %, Fe, Si, Cu, Zn total 0.5 wt. %) were roughened in a hydrochloric acid bath using an alternating current (concentration of HCl, c = 10 g dm^{-3}, current density, j = 10 A dm^{-3}, duration, t = 4 min, temperature of the electrolyte, τ = 22 ºC).[7]

Before cementation, the electrochemically roughened aluminum specimens A-1, A-2, A-3, A-4 were treated by the following procedure:

– removal of grease using a solution of "Meripol 45" (Merima, Kruševac), concentration 1 wt. %, at 25 ºC, for 3 min;

– washing with distilled water

– treatment with 18 wt. % HNO_3 acid for 90 s at 25 ºC, in order to remove aluminates and intrusions (Fe, Si, Zn and Cu) from the aluminum surface, and

– washing with distilled water.

The 3 × 3 cm specimens A-1, A-2, A-3 and A-4, prepared by this procedure, were immersed in the solutions given in Table I. The solutions were prepared by first diluting NaOH or commercial developer (ZIP, Kruševac), and then chromium chloride was added. The commercial developers for positive offset printing plates consist mostly of concentrated aqueous solution of NaOH, in order to dissolve exposed part of the photosensitive layer. The sample A-5 was neither treated by the procedure for removing grease nor by the developing procedure.

TABLE I. Composition of the solutions and the conditions of treatment of the aluminum specimens

Specimen	Composition of aqueous solution	Temperature	Duration
A-1	NaOH; 10 g dm^{-3}; $CrCl_3$; 5 g dm^{-3}		
A-2	HCl; 10 g dm^{-3}; $CrCl_3$; 5 g dm^{-3}		
A-3	Positive developer for printing plates diluted by water to concentration of 20 % vol. $CrCl_3$ 5 g dm^{-3}	23 – 25 ºC	4 min
A-4	NaOH 10 g dm^{-3}		
A-5	Specimen that was not treated chemically		

Cementation was made from commercial developer as the alkaline medium in order to test the possibility of electroless chemical plating under real conditions of the reprographic process in printing shops.

The chromium-treated specimens together with the non-chromium-plated specimens were examined by scanning electron microscopy (SEM) using the EDAX procedure (energy disperse X-ray detector). The measurements was conducted in three different laboratories:

– Vinča Institute of Nuclear Sciences, Belgrade (Phillips scanning electron microscope),

– Functional Materials, Saarland University, Saarbrücken (Cam Scan FE scanning electron microscope) and

– Fraunhofer – Institut Produktionstechnik und Automatisierung, Stuttgart (Cambridge 360 scanning electron microscope) in order to obtain independent confirmation of the validity of the cementation process. A qualitative and quantitative analysis of the surface layer was made in order to confirm the possibility of obtaining a chromium layer by this method. This SEM method shows the composition of a 2 μm deep surface layer.[8]

The spectral reflection characteristics of the chromium-treated and non-chromium treated specimens were also determined. The measurements were recorded using a narrowband spectrophotometer (Spektrolino, Gretag). This spectrophotometer measures the reflectance of visible light (380–730 nm) in bands 10 nm wide. The incident light arrives at the specimen at an angle of 45º, and the reflectance is measured at 0º.

The influence of chromium on the wettability of the non-printing elements of the printing form was examined by drop spreading. Specimens A-1 and A-4 (six pieces of each) were examined by dropping distilled water in drops onto the surface of a specimen. As a base for preparing the specimens, a previously roughened aluminum foil 0.1 mm thick was used. This is the normal type of foil employed for making printing plates. The average roughness of this foil was $R_a = 1.5$ μm.[9]

The dropper was situated 5 mm above the surface. Dropping proceeded under gravitational force. The drop volume was determined by counting the number of drops, $n_d = 300$, necessary to fill $V = 3.3$ cm^3. The drop volume V_d, in this experiment was calculated as $V_d = V/n_d = 11$ mm^3.

The minimal distance between the dropper and the surface was selected to minimize the influence of physical factors (*i.e.,* kinetic energy) on the drop spreading.[4]

Only one drop was dropped onto the surface of a specimen. A picture of the drop was taken by a digital camera Olympus C-4040, 4.1 Megapixel. The pictures were made from two directions, in order to measure the diameter of the spread drop (a measuring gauge was recorded together with the drop) and the contact angle.

If the surface is homogeneous and equally prepared in all directions, the drop forms a centrally symmetrical shape. If the dampening solution forms asymmetrical or dendritic shapes it might mean that the surface had not been prepared well and it could carry grease, impurities, oxides and/or deposits that change the surface properties of the base material. In this work, asymmetrical figures were not measured.

Each picture of a spread drop with the measuring gauge was computer analyzed using CorelDRAW software. The diameter of the circle was measured using this software with an accuracy of ±0.1 mm.

The same software was used for measuring the contact angle. The picture of the profile of the spread drop was imported into the software and a tangent to the drop profile was plotted. The contact angle was measured by this software with an accuracy of ±1°.

Finally, printing tests were performed on printing forms that had been made under the same conditions as the specimens A-1 and A-4.

In order to make the printing conditions severe, both printing forms consisted of non-printing elements only. The samples were printed simultaneously using a printing machine ADAST Romayor 313, equipped with a non-alcohol-dampening unit. Usually the mounted printing forms are treated with acid solutions of gum arabic in order to increase the wettability of the non-printing elements; this was not performed in these experiments. After every exchange of printing test conditions, control prints were taken (after 500, 1000, 2000, 2500 and 3500 prints). The control prints were made on 80 g/m^2-offset paper, with the printing ink Hartmann black and the dampening solution "Jetka", Cinkarna Celje (Slovenia).

At the beginning of the test, the ink flow and the dampening solution flow were set to the usual values and control prints P-1-500 (from printing form A-1, print No. 500) and P-4-500 (from printing form A-4, print No. 500) were taken.

In order to avoid the influence of inking and dampening unit imperfections, the positions of the chromium and non-chromium treated samples on the form cylinder were exchanged and control prints P-1-1000 and P-4-1000 were taken.

The ink flow was set to maximum and the dampening solution flow was set to minimum in order to make the printing conditions as severe as possible. The control prints P-1-2000 and P-4-2000 were taken.

The positions of the samples on the form cylinder were exchanged again and the control print P-1-2500 and P-4-2500 were taken.

The last control prints, P-1-3500 and P-4-3500, were printed under extremely unfavorable printing conditions, with maximum ink flow and no dampening solution flow (the dampening unit was switched off).

The purity of the prints made from different samples (printing forms) was analyzed and the results were taken as a measure of the wettability and convenience of the printing forms.

The purity of the prints made by the two types of the printing forms were analyzed in two ways:
– based on densitometric measurements and
– based on statistical analysis of scanned prints.

The optical density of the prints was measured using the narrowband spectrophotometer Spektrolino, Gretag. Fifteen measuring positions in the middle part of each print were chosen, in order to avoid any influence of eventual damages in the border zone. The optical density, D, was measured on three samples from each series of control prints, made in different stages of the experimental printing. The optical density was defined as

$$D = \log I_0/I \qquad (2)$$

where I_0 is amount of incident light and I is amount of reflected light. The average value of optical density for every series of the prints was calculated.[10]

One control print from each series was scanned using a flatbed scanner HewlettPackard 7400c. The scanning resolution was 100 dots per inch (dpi) and the size of the scanned field was 4.00×9.00 inches (10.2× 22.9 cm). In order to avoid the influence of damage in the border zone, the zone in the middle of the print was

scanned. All specimens were scanned as grayscales, and 8-bit bitmaps were obtained. The whiteness of each spot is expressed by a value from zero (spots that are recognized as black) to 255 (spots that are recognized as white). So, there are $2^8 = 256$ steps of whiteness in such bit-maps.

Statistical analysis of the scanned bit-maps was performed using a program package Math-Lab 5.0.[11] Each bit-map was converted into a matrix composed of numerical values of each scanned spot. There were $4.00 \times 100 \times 9.00 \times 100 = 360,000$ spots (*i.e.*, numerical values) in each bit-map. The shape of each spot was square 0.254 mm in size. The matrixes were converted into vectors, in order to enable the softwere to sort and count spots according to their whiteness. As a result histograms were obtained, which represent the distribution of spots according to their whiteness.

Furthermore, the total whiteness was calculated as

$$w = \sum_{i=0}^{255} n_i\ i \tag{3}$$

where i is the number representing the whiteness value of an analyzed spot an n_i is number of spots in the analyzed bit-map with same whiteness value i. For example, the w-value for totally white 4.00×9.00 inches surface scanned at 100 dpi, calculated by Eq. (3) would be $w_{white} = 360000 \times 255 = 918 \times 10^5$, and for totally black $w_{balck} = 360000 \times 0 = 0$.

RESULTS AND DISCUSSION

The results of the qualitative and quantitative analysis of the surface layers of the samples A-0, A-1, A-2, A-3, A-4 and A-5, conducted at the Vinča Institute of Nuclear Sciences are shown in Table II. The results of the qualitative and quantitative analysis of the surface layers of the samples A-1 and A-4, conducted in Functional Materials, Saarland University, and Fraunhofer – Institut Produktionstechnik und Automatisierung, are shown in Table III.

TABLE II. Resuilts of the quantitative and qualitative analysis of surface layer (measured in the Vinča Institute, Belgrade)

Element	wt. % (at. %) of chromium				
	A-1	A-2	A-3	A-4	A-5
O	18.95 (28.43)	6.09 (9.88)	15.29 (23.49)	21.72 (32.06)	23.29 (34.00)
Al	79.59 (70.80)	92.95 (89.51)	82.36 (75.05)	76.02 (66.52)	74.66 (64.61)
P	– (–)	0.28 (0.16)	0.47 (0.37)	0.27 (0.21)	0.44 (0.33)
S	0.22 (0.16)	0.15 (0.12)	0.26 (0.20)	– (–)	– (–)
Cl	– (–)	– (–)	0.44 (0.30)	1.48 (0.99)	1.60 (1.06)
Ca	0.33 (0.20)	0.20 (0.13)	0.32 (0.20)	– (–)	– (–)
Cr	0.48 (0.22)	– (–)	0.47 (0.22)	– (–)	– (–)
Fe	0.43 (0.43)	0.35 (0.16)	0.39 (0.17)	0.44	– (–)

No chromium was evidenced in the surface layer of the control specimen A-5. The specimen A-5 represents the aluminum foil that is used as a base for preparing the chro-

mium-plated specimens. Also, the surface layers of the specimen A-2, which had been treated in the acid solution and the specimen A-4, which had been treated in the alkaline solution, but without chromium ions, contained no measurable quantity of chromium. The spcimen A-4 represents the control specimen for A-1.

The presence of chromium was confirmed in specimens A-1 and A-3, which had been treated in alkaline solutions of chromium chloride.

Furthermore, the presence of chromium was confirmed in specimen A-1 that had been tested in the other two laboratories.

TABLE III. Chromium content on specimens A-1 and A-4 (measured in the Functional Materials, Saarland University, Saarbrücken and Fraunhofer Insitute, Stuttgart)

wt. % (at. %) Of chromium measured in Functional Materials, Saarland University		wt. % (at. %) Of chromium measured in Fraunhofer Institute, Stuttgart	
A-1	A-4	A-1	A-4
0.54 (0.28)	– (–)	0.25 (0.13)	– (–)

As aluminum very easily forms an oxide layer with atmospheric oxygen, cementation on the aluminum could not occur unitl the oxide layer had been removed, as in the case of zincate or stanate (Alstan) treatment. A solution of NaOH was used to remove the oxide layer and uncover the pure aluminum surface, which enabled the start of the cementation on the aluminum. It is assumed that the cementation ceases when the entire aluminum surface had been covered with chromium.

Cementation cannot take place from acid solutions, because the Cr^{3+} ion is highly hydrated and so is very hard to reduce. In addition, chromium dissolves in HCl.

The dependence of the spectral reflection on wavelength is shown in Fig. 1. The dashed line represents the chromium-treated specimen, and the solid line represents the non-chromium-treated specimen. The diagrams shows average values obtained from three measurements.

The specimens on which chromium was found had a different color to the non-chromium-plated specimens. Spectral reflection analysis showed a significant difference between specimens treated in alkali solutions with and without chromium ions. The specimen treated with chromium ions reflected about 10 % more light than the non-treated specimen. Both specimens were neutral, gray, and the reflectance was almost independent of wavelength. It can be assumed that the increase in reflectance is due to the chromium deposit on the surface.

The average diameter of the spread drop, as well as the average value of the contact angle for specimens A-1 and A-4, from six measurements, are shown in Table IV.

The same amount (one drop) of H_2O covers approx. 3/4 more area of the roughened aluminum specimen if it is chromium-treated. Reducing the amount of dampening solution in the offset printing process is very important, because of ink/water balance, dimensional stability of the printed paper, economy and environment protection.

The contact angle with H_2O on chromium-treated specimens is approx. 2/3 of the contact angle on non-chromium-treated specimens.

TABLE IV. Average values of the diameter of the spreaded drop and average value of the contact angle on specimens A-1 and A-4

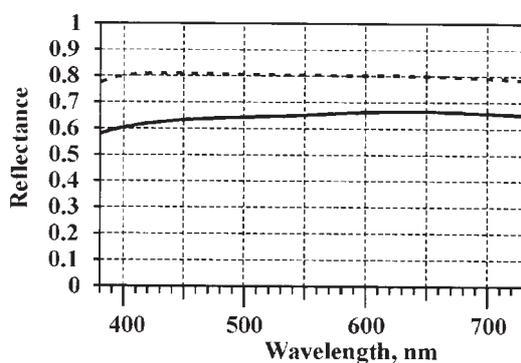| Average diameter (wetted area/mm^2) of spreaded drop/mm		Average contact angle with H$_2$O/°	
A-1 (Chromium treated)	A-4 (Non-chromium-treated)	A-1 (Chromium treated)	A-4 (Non-chromium-treated)
5.8 (26.4)	4.4 (15.2)	38	59

Fig. 1. Dependence of the reflection on wavelength: (- - -) specimen A-1; (—) specimen A-4.

The visual comparison of the wettability of the chromium-treated and non-chromium treated specimens are shown in Fig. 2. The measuring scale with 0.1 mm gradation in Fig. 2 a and b was added using software. The lines representing the contact angle of water on the surface of the specimen was also added using software. The pictures taken with the same enlargement show obvious differences in the wettability of the chromium-treated and non-chromium-treated specimens.

At the beginning of the experimental printing, the flows of ink and dampening solution were set to normal values for this type of printing machine. Visual analysis of the prints demonstrate that intensive toning occurred on the prints made from the non-chromium-treated control printing form, while the prints from the chromium-plated printing form were clear. The same behavior of the non-chromium-plated and chrome-plated printing form was obtained in the other two series of printing tests.

Even after changing the printing conditions (position of the printing forms on the form cylinder, reducing the flow of the dampening solution and increasing the ink flow, setting the ink flow to maximum and the dampening solution flow to minimum, another exchange of the position of the printing forms on the form cylinder, setting the dampening solution flow to zero) the control prints from the chromium-plated printing form were much clearer than the control prints made from the non-chromium-treated printing plates. The control prints from non-chromium-treated printing plates always suggested that toning had occurred.

The results of the quantitative analysis of the purity of the prints made from printing forms A-1 and A-4 are shown in Table V.
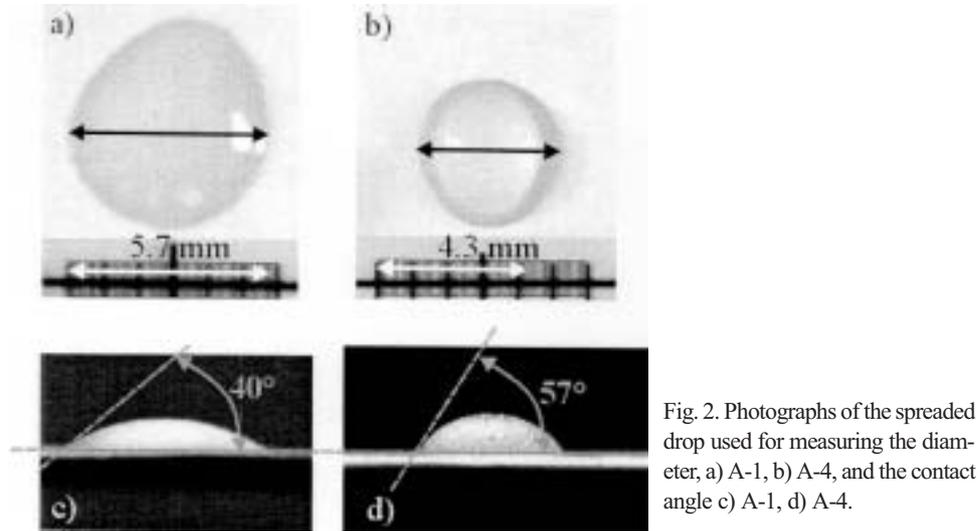
Fig. 2. Photographs of the spreaded drop used for measuring the diameter, a) A-1, b) A-4, and the contact angle c) A-1, d) A-4.

TABLE V. Average values of density and whiteness of the control prints

Control print No.	P-1-500	P-4-500	P-1-1000	P-4-1000	P-1-2000	P-4-2000	P-1-2500	P-4-2500	P-1-3500	P-4-3500	Paper
Density average value	0.07	0.09	0.06	0.08	0.06	0.08	0.06	0.08	0.09	0.12	0.05
Total whiteness × 10^{-5}	901	876	906	893	908	896	911	895	877	846	917

Both method of analysis of the control prints showed that the control prints made from the chromium-treated specimens are purer than the prints made from the non-chromium-treated specimens. The density measurements showed that the optical density of the
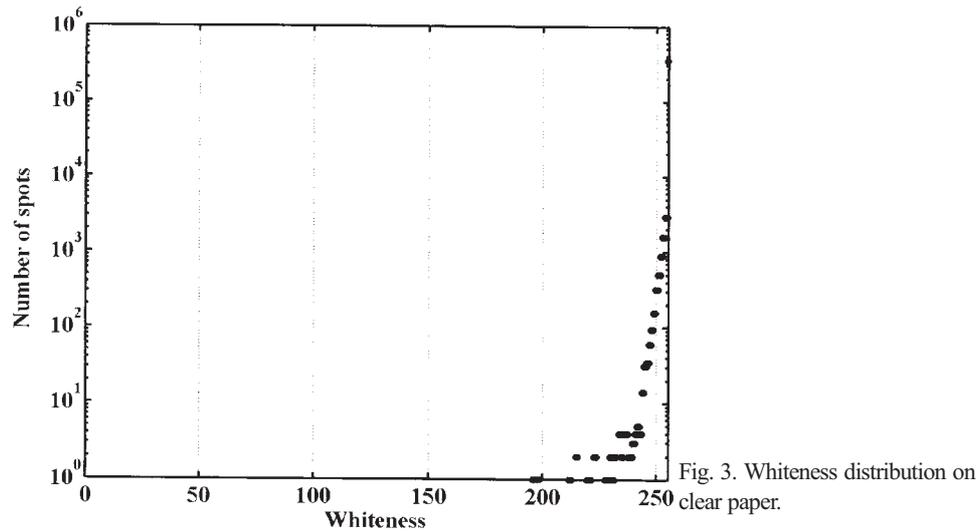

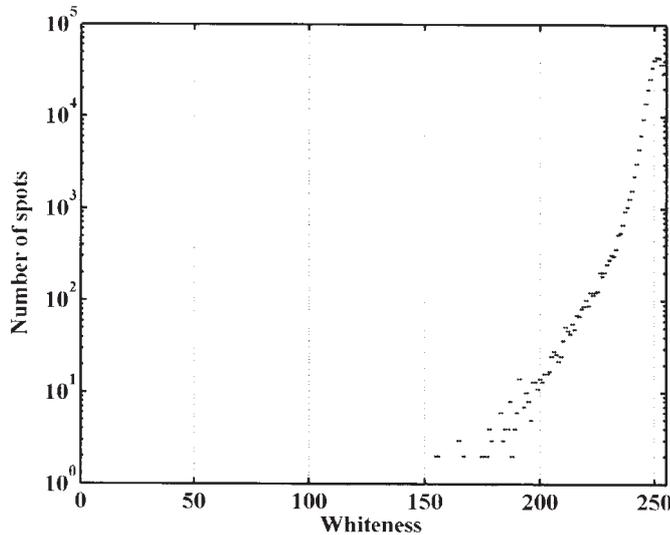Fig. 3. Whiteness distribution on clear paper.

Fig. 4. Whiteness distribution on control print P-1-500.

chromium-treated prints is 22–25 % lower than the optical density of the non-chromium treated specimens. The results of calculating the total whiteness by Eq. (3) confirm that the control prints made from the chromium-treated printing forms are purer (whiter) than the control prints made from the non-chromium-treated samples.

Figure 3 shows the distribution of whiteness on the unprinted paper. The diagram for an ideally white surface would have only one point, *i.e.*, all spots would have the whiteness value $w = 255$. The paper is not ideal and there are spots that are not totally white, but the distribution is concentrated on the right side of histogram. The minimum w-value is approx. 200.

Figures 4 and 5 show typical distribution of the whiteness of control prints made in this work. The diagram for the control print P-4-500 which was printed from the non-chro-
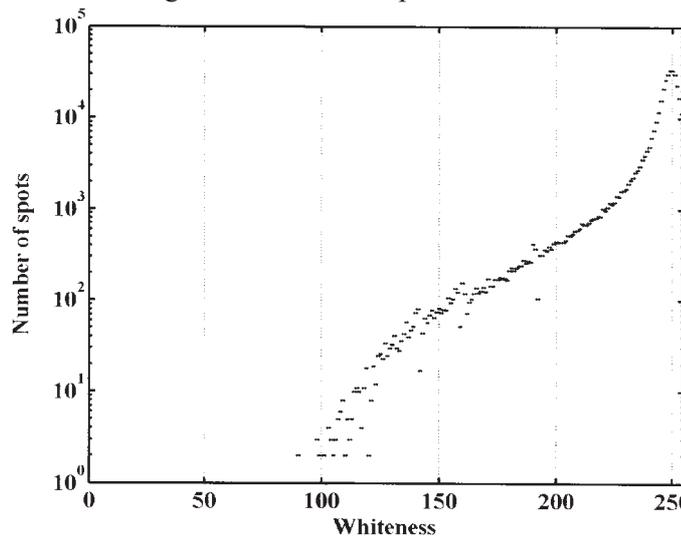

Fig. 5. Whiteness distribution on control print P-4-500.

mium-treated printing form shows that there are many spots with a whiteness value smaller than 150, and there are even a few spots with a whiteness value smaller than 100. On the control print P-1-500 that was printed from the chromium-treated printing form there are no spots with whiteness value smaller than 150. This means that with the same dampening solution flow the chromium-treated printing form repels the printing ink better than the non-chromium-treated printing form. Considering that all the conditions of making the printing forms and printing conditions, except the electroless deposition procedure were the same, it could be concluded that an electroless deposited layer of chromium improves the printing properties of an offset printing form, because of the increased wettability.

CONCLUSION

Based on the performed experiments, it can be concluded that cementation of chromium onto aluminum from alkaline solutions at room temperature is possible.

Comparing the wettability of the examined samples, electroless deposition of chromium increases the water wettability of aluminum. The contact angle with water is reduced by 1/3 by electroless deposition of chromium on roughened aluminum. The area wetted by the same amount of the same dampening solution is bigger on the chromium-treated samples by 3/4.

With the same dampening solution flow and the same ink flow using the same printing machine, the chromium-plated printing form gave clearer prints than the non-chromium-treated printing form, because of the increased wettability. The average otpical density of control prints made from the chromium-plated printing form is approx. 25 % lower than the control print made from the non-chromium-treated printing form. The greater purity of the control prints made from the chromium-treated printing form is confirmed by analysis of the total whiteness of the control prints.

This study showed that electroless chromium plating could be applied under the same condition as in real production, so the method is simple and suitable.

The kinetics of cementation of chromium onto aluminum should be examined in a further investigation, as should the structure of the surface layer.

ИЗВОД

УТИЦАЈ ХРОМА ИСТАЛОЖЕНОГ ЦЕМЕНТАЦИЈОМ НА ШТАМПАРСКА СВОЈСТВА ФОРМЕ ЗА ОФСЕТ ШТАМПУ

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Приказан је метод за побољшање својстава штампарских форми за офсет штампу. Узорци техничког алуминијума су електрохемијски нахрапављени и третирани различитим растворима у циљу испитивања могућности хемијског таложења хрома на алуминијуму. Састав површинског слоја обрађиваних узорака одређен је EDAX-ом. Присуство хрома доказано је на узорцима који су третирани алкалним растворима који су садржали Cr^{3+} јоне, док на узорцима који су третирани растворима без присуства хромних јона присуство хрома

није потврђено. Измерена је спектрална рефлексија хромираних и нехромираних узорака. Извршено је поређење квашљивости хромираних и нехромираних узорака. Квашљивост нехромираних узорака упоређена је са квашљивошћу хромираних узорака мерењем контактног угла са водом као и оквашене површине. Констатовано је да се хромирани узорци боље квасе од нехромираних. Експериментално штампање је извршено под реалним условима. Котролни отисци анализирани су на основу дензитометријских мерења и статистичком анализом скениране слике. Контролни отисци начињени помоћу хромираних форми били су чистији од контролних отисака начињених нехромираним формама под свим условима штампања, а посебно у отежаним штампарским условима.

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