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ELECTRODEPOSITION OF CHROMIUM WITH PERIODIC REVERSE AND PULSED CURRENT

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This report covers an investigation of the effects of periodic reverse current and pulsed direct current on properties and metal distribution of chromium electrodeposits. The plating current was reversed for short periods of various lengths during deposition of chromium at various current densities and in baths at several concentrations of chromium trioxide or CrO_3. Several chemicals were added to the standard bath in an effort to further improve

20. ABSTRACT (continued)

deposit distribution during periodic reverse (PR) plating. Several of the chemicals tried as well as the lower bath concentrations were effective in improving distribution in addition to the improvement obtained with PR. Thickness ratios of less than one were obtained on some panels and screw threads were plated with a ratio as low as 1.3 using PR. Small but definite improvements in the distribution of chromium deposits were obtained using pulsed dc current with the most uniform thickness obtained in the dilute bath. Also, nearly all deposits from the dilute bath were bright over a range of pulse times. (U) (Young, John P.)

FOREWORD

This report was prepared by John P. Young of the Corrosion and Electrodeposition Section, Metallurgy Division, Institute for Materials Research, National Bureau of Standards, Washington, DC, in compliance with MIPR A1-3-65501-M2-W9. This work was performed for the Research Directorate, GEN Thomas J. Rodman Laboratory, Rock Island, Illinois with Donald H. Sale as Project Engineer.

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INTRODUCTION

Some work has been done using periodic reverse (PR) current for plating chromium with resulting claims for improved adhesion, deposit distribution and greater density of the deposit, etc. The work is detailed in items listed in the bibliography at the end of this report. PR is effective in producing deposits of uniform thickness with little edge build-up and with smooth, bright surfaces from copper, silver, and gold cyanide baths. It has been used with only a few other plating solutions. The deposition of chromium results in excessive build-up of the deposit on edges and corners and on areas nearest the anode. This work investigated various combinations of forward and reverse current cycles and pulsed current (PC) times and levels to improve chromium deposit thickness distribution (see Appendix for tabulated data).

There are several differences in plating from a chromic acid bath and from the cyanide baths that are used successfully with PR and PC. The cyanide baths tend to form a resistant cathode layer which becomes more resistant the higher the current density. Chromium is more efficiently deposited at higher current densities. Due to the generally low efficiency (12-15%) of chromium deposition, there is a copious release of hydrogen which causes considerable agitation at the cathode surface. This condition does not occur in most cyanide baths at normal plating conditions. Also, the cyanide baths are alkaline and mildly oxidizing while the chromium bath is strongly acid and strongly oxidizing. The great dissimilarity of the two gives no hint for the successful PR plating of chromium.

Our work revealed that when the chromium deposit was made the anode (i.e. current reversed) it dissolved with 100 percent efficiency as hexavalent chromium. At plating voltage the reverse current was 2 to 3 times greater. The results of anodic current-potential tests by Dr. Bertocci of this laboratory are shown in Figure 1.

This disparity in efficiency between the forward and reverse parts of the PR cycle necessitated a reduction in current or time or both to at least one-sixth of that for the forward part of the cycle. It was found that short PR cycles of less than one minute total duration which are effective in the cyanide baths could not be effectively used especially in the dilute chromium baths because of poor distribution and the dark and rough deposits obtained for most short cycle combinations tried. Cycles longer than three minutes including a reverse portion long enough to be effective in improving distribution caused removal of lead peroxide from the anode and the formation of lead chromate which built up in the bath as a sludge and interfered with the efficiency of deposition and the formation of a smooth deposit.

Most of the PR cycles tried within the limits of the conditions outlined above resulted in chromium deposits with no better or poorer distribution than that obtained without PR. In this report data are given on those cycles and conditions giving the best results, for the most part, and that might have practical use. The obvious goal of the experiments is to obtain a thickness ratio (edge-to-center ratio) of one. Therefore, the success of any particular test may be judged by how close the ratios are to this figure in comparison to the best ratios obtained without PR.

The addition of certain chemicals to the chromium bath improved deposit distribution both with and without PR. Additives tried with some success included oxalic acid, boric acid and sodium hydroxide. The more dilute chromium plating baths containing 100 or 250 g/l of CrO_3 gave slightly better distribution on the test panels than more concentrated solutions. Also, the additives were more effective in the dilute solutions with and without PR.

The anticipated poor adhesion between chromium layers formed during PR plating did not occur. It was therefore not necessary to use special techniques such as slow build-up of the current before each forward part of the cycle. In fact, no layering was visible in cross-sections of the deposits until points of very high current density were examined such as chromium deposited using PR on the tops of threads.

The use of PR, dilute chromium plating solutions and additives to the plating solutions individually and collectively resulted in improved edge-to-center distribution of chromium deposited on the flat test specimens used. PR cycles used most successfully consisted of from 1 to 9 seconds reverse current depending on forward time and the amount of reverse current. Forward times of from 60 to 180 seconds gave the most consistent good results with 120 seconds perhaps the most effective. Reverse currents from one-half to double or triple the forward current but with equal ampere seconds were effective. Equal or higher reverse current was effective with low forward current and short cycles. Edge-to-center thickness ratios of 1.0 or less were sometimes obtained under several conditions whereas without PR or additives the ratio could be 6 or 8. No full bright deposits were obtained using PR. See Figure 2 for several SEM pictures of the as-plated surface. Hardness values of all PR deposits were one-fourth to one-third less than the 1000 to 1100 Knoop hardness number for bright, hard chromium.

A current supply was obtained and tests were made using a pulsed dc current in several concentrations of the chromium bath. Pulse peak levels and durations were varied as were base current levels and intervals between pulses. Several average current density levels for the pulse on-off cycle and bath temperatures were tried. The literature (see bibliography) indicates improved hardness, better distribution and improved brightness for chromium deposits plated using pulsed current or PC. A slight improvement was realized in deposit distribution and brightness under certain conditions of pulse ratios and bath concentration. Hardness was not improved, at least at a bath temperature of 65°C.

EQUIPMENT

The experimental chromium plating baths were contained in a one liter beaker immersed in a water bath containing a copper heating-cooling coil through which steam or cold water could be passed to maintain the desired plating bath temperature. This assembly was placed on a magnetic stirrer used continuously during all plating runs to maintain a uniform temperature and concentration throughout the plating bath. (See Figure 3).

Both a filtered and an unfiltered full wave rectifier were tried as a plating current source without any difference in results being observed. Nearly all runs were made using the filtered power supply with the unfiltered source connected to the circuit when additional current or voltage was needed for high current tests in dilute solution. The current reversing solenoid switch was a standard 100 ampere unit that allowed insertion of current controlling resistance in either the forward or reverse portions of the current cycle. Duration of each phase of the cycle was controlled by a tandem recycling timer made by the Industrial Timer Corporation. For very high reverse (three or four times forward) currents it was necessary to use a resistance in the forward part of the cycle. It would have been possible to supply each phase of the PR cycle with separate rectifiers but it was not done in these tests.

Thickness of the deposits was measured at the extreme edges and near the center with a micrometer. Measurements by a Kocour thickness tester were also made on the deposits plated with PR current but about 1/16 to 1/8 inch from the edge depending on edge build-up. This instrument uses a sulfuric acid solution to dissolve the chromium electrolytically with thickness proportional to time. It requires the seating of a rubber grommet on the surface of the deposit to contain the electrolyte. Thickness and distribution was also checked on some specimens by sectioning, mounting in plastic and microscopical examination. A scanning electron microscope was used for inspecting the surface texture of a few of the specimens.

PROCEDURE

The specimens used were mild steel panels about thirty mils thick and three inches square. They were stopped off with insulating lacquer on the side away from the anode. The only surface treatment was degreasing before applying the lacquer. The specimens were supported at the center on a one-quarter inch diameter rod bent at a right angle at one end. The specimen was secured with a nut and washers. All exposed areas of the support were stopped off. The anode was a 3/16 inch thick lead sheet slightly larger in area than the specimen and was drilled with about three one-quarter inch holes per square inch of surface. A number of runs were made with the specimens at an angle of 30 to 35 degrees to the anode to increase deposit distribution differentials but most tests were made with the electrodes parallel and separated by about 13/16 inch. This separation was maintained constant by a plastic spacer which screwed onto the exposed end of the supporting rod and bore against the anode surface. See Figure 3.

The specimens were treated before plating by a reverse current of one to three minutes duration in the plating bath with an additional forward current treatment below plating level for pulse-plated panels only. Those that were to be plated by PR current were first plated three to five minutes with forward current only, before the PR was started. After plating from one to two and one half hours depending on the current density and PR cycle used, current was shut off at the end of a forward cycle which gave better appearance of the deposit surface. The specimens were rinsed in warm water and the stop-off peeled from the back. Micrometer measurements were made at the edges and near the center. Chromium thickness was determined by subtracting the known thickness of the steel. The coulometric thickness measurements were made on the PR plated panels only, in accordance with the procedure for using the Kocour thickness tester. The sulfuric acid electrolyte furnished by the instrument manufacturer was used. The ratio of edge-to-center distribution of the deposit was obtained by averaging the thickness of two edges of the specimen and dividing this by the thickness at the center. Ratios for both coulometric and micrometer measurements were calculated and included in the data for PR deposits; micrometer data only, for PC deposits.

Deposits that were to be measured for hardness and for examination were sectioned at the mid-point of one edge for a distance of about three fourths inch toward the center. These approximately one fourth inch wide sections were mounted on edge in bakelite in a standard mounting press.

The plating solutions were all made up with technical grade chromic acid and with sulfuric acid as the catalyst at a chromic-sulfuric acid ratio of one hundred to one. Solutions were used for several runs each by maintaining the desired chromic acid concentration. Runs were made for comparison without PR in the various bath concentrations and compositions tried and at the various plating conditions. The 532 tests were necessarily of an exploratory nature and complete series were not necessarily made of each variable if it did not look promising. This method of testing makes absolute comparison between variables not always possible but allows broader coverage of the enormous number of possibilities with limited finances and time.

EXPERIMENTAL RESULTS WITH PR

Cycle-time length tests

Initial experiments showed much less differential between edge and center deposit thickness than expected using parallel electrodes so the first group of PR tests were made with the cathode at an angle of 30-35 degrees to the anode to accentuate distribution disparity. The closest edge was one-fourth inch from the anode.

The first tests using PR used varying cycle times to try and determine the most beneficial cycle length and proportion of forward time to reverse time. It was found that the forward cycle had to be more than six times longer than the reverse cycle or no deposit was formed. The dissolution efficiency during the reverse was about six times that of the plating efficiency during the forward part of the cycle. For most conditions the effective cycle (the resultant forward current-time total per cycle) had to be in the order of ten to twenty times the reverse total to obtain coverage of the specimen by the deposit.

Short forward times of much less than sixty seconds resulted in dark, matte deposits while longer forward times up to 180 seconds gave a lighter, smoother deposit although the effective cycle was the same. This can in part be attributed to the fact that all runs were stopped at the end of the forward time and a longer period under normal bright plating conditions resulted. But the improved appearance was greater than this effect could account for. It may be that the longer cycles allowed more uniform dissolution of the plated surface rather than just enlarging the ever-present channels and cracks as the short reverse times may do. It could also be related to the time required to form a uniform cathode film or layer.

The cycles checked varied from six to 180 seconds forward and one-fourth to ten second reverse. Those cycles that gave a deposit distribution ratio (coulometric measurement) of less than two on the angled cathode in the 200 g/l CrO_3 bath were:

Forward - Reverse Time	Effective Cycle	asd (Forward-Reverse)	Surface	Edge to Center Thickness
18-1/8 sec.	96-1	+20, -30	Matte	1.9 ratio
60-1	20-1	+20, -60	Satin	1.7
180-1	60-1	+20, -54	Semi-brite	1.9

The average ratio was 3.3 for non-PR deposits at 20 A/dm^2(asd) and range from 2.2 to 6. A test of cycle lengths in the 500 g/l CrO_3 bath with parallel electrodes and at reverse current levels one-half forward current resulted in ratios averaging 1.4. The cycles giving ratios of 1.3 or less were:

Forward - Reverse Time	Effective Cycle	asd (Forward-Reverse)	Surface	Edge to Center Thickness
60-6 sec.	20-1	+30, -15	Satin	1.3 ratio
90-13.5	13-1	+30, -15	Satin	1.1
90-9	20-1	+30, -15	Satin	1.3
90-4.5	40-1	+30, -15	Satin	1.2
120-12	20-1	+30, -15	Satin	1.2
120-6	40-1	+30, -15	Satin	1.3
120-3	80-1	+30, -15	Satin	1.3
180-18	20-1	+30, -15	Satin	1.2
180-9	40-1	+30, -15	Satin	1.3
180-4.5	80-1	+30, -15	Satin	1.3
150-15	20-1	+30, -15	Satin	1.3

Two runs at 30 asd without PR had an average ratio of 2.3. As mentioned earlier, runs with forward times of less than 60 sec. and effective ratios less than 10-1 result in poor quality deposits and runs with reverse times of more than about 20 sec. caused anode sludging.

A review of the overall performance of the cycles used at various current densities with and without additives in the bath throughout this investigation showed several that resulted in ratios of 1.3 or less more consistently than when no PR was used. The following indicates the most likely level of reverse current and the percentage of all the tests for which a 1.3 or better ratio resulted:

No PR	44%	
180-9 sec.	86	equal reverse (to forward current)
60-3	70	equal reverse
120-3	68	high reverse
180-6	63	equal or high reverse
120-6	63	low or equal reverse
60-1-1/2	60	high reverse

Bath Concentration

The initial test of the effect of the concentration of chromic acid in the bath was made with the cathode at an angle to the anode. Concentrations of 300, 400, and 500 g/l CrO_3 were tried. Ratios obtained from this test were not optimum because of the short cycle times used. At the time the test was run, the effect of cycle length was not fully appreciated. The comparative performance for a more or less similar range of cycles and plating conditions at each bath concentration is shown below. The best average distribution (coulometric) is indicated as one.

300 g/l CrO_3 - 0.45
400 - 1.2
500 - 1.9

Performance for several series of test with a parallel cathode under conditions that are comparable to the above data and calculated the same way was:

100 g/l CrO_3 - 0.45
250 - 0.45
500 - 0.62

Data for a few tests selected for similar plating conditions for the 500 and 100 g/l CrO_3 baths are shown below:

			Thickness Ratio			
	Effective	CD	100 CrO_3		500 CrO_3	
PR Cycle (sec)	Cycle	(asd)	Kocour	Micrometer	K	M
none	-	50	1.2	1.5	1.8	4.3
none	-	30	1.3	1.9	2.8	7.3
none	-	20	1.5	2.9	1.6	5.2
120-1	40-1	+30-90	1.3	1.7	1.6	5.7
120-6	40-1	+30-15	1.3	2.7	1.3	6.5
40-4 (45-4.5)	40-1	+50-25	1.8	1.8	1.8	7.5
60-6 (90-9)	20-1	+50-25	1.3	1.4	1.7	6.8
60-3 (60-4)	20-1(15-1)	+30-30	1.4	2.1	1.3	3.1

NOTE: Parentheses indicate differences in cycle for 500 g/l bath.

These tests of bath concentration showed that the more dilute baths gave substantially less edge build-up on flat specimens with and without PR. Improvement in distribution by the use of PR is not evident in the above detailed data for either the dilute or concentrated baths.

Figure 4 illustrates deposit thickness ratio differences induced by bath concentration. Cross sections of deposits both with or without PR show differences in thickness ratios resulting from bath concentration as indicated in Figures 5 and 6.

Effect of Coating Back of Specimen

A test was made to check the amount of edge and corner build-up and the amount of coverage of the back area (facing away from the single anode) by the chromium while using PR under various conditions. This might give a better idea of differences in throwing power. The tests were run in the 500 g/l CrO_3 bath.

The backs of the panels were completely covered with chromium when no PR or the less sacrificial PR cycles were used. As might be expected a less sacrificial PR cycle was required to eliminate bare areas on the front of the panels with the backs uncoated than when coated, and the deposit build-up at the extreme edges of the panel was diminished. However, improved deposit distribution over the entire front of the panel was not realized. Some thickness ratios for conditions that resulted in complete coverage of the front of the panels with uncoated backs compared to those with coated (c) backs follow:

PR Cycle	Effective Cycle	CD	Surface	Thickness Ratio
No PR (c)	-	+30 (asd)	semi-brite	1.8
No PR	-	+30 (asd)	light matte	1.5
60-1 (c)	30-1	+30 (asd)	light matte	1.5
60-1	30-1	+30 (asd)	satin	1.0
90-1 (c)	45-1	+30 (asd)	light matte	1.5
90-1	45-1	+30 (asd)	satin	0.9
90-2 (c)	22-1	+30 (asd)	light matte	1.5
90-2	20-1	+30 (asd)	satin	1.1
180-4 (c)	23-1	+30 (asd)	satin	1.7
180-4	22-1	+30 (asd)	satin	1.3
180-6 (c)	15-1	+30 (asd)	satin	1.3
180-6	15-1	+30 (asd)	satin	0.9
45-1 (c)	22-1	+30 (asd)	satin	1.4
45-1	22-1	+30 (asd)	light matte	1.4

Average ratios were 1.2 for uncoated and 1.5 for coated specimens. The difference between the averages 1.2 and 1.5 is about the same as between the sample with no PR, 1.5 and 1.8, indicating that the benefit of PR is about the same in each case. The tests were made in the 500 g/l CrO_3 bath.

An interesting side effect of the plating of panels with uncoated backs was the formation of rainbow colored oxides in areas of current density too low for deposition of chromium. It presumably formed during the reverse part of the cycle. This oxide coating was fairly durable and the colors persistant.

Current Density Tests

A determination of the effect of several plating current densities on PR chromium distributions was made in the 500 g/l CrO_3 bath. A range of PR cycles were checked for each current density. All deposits on each average were made with short reverse times and high reverse cd. The following is a summary of the ratios obtained for each current value:

20 asd forward current	1.6 coulometric	2.2 micrometer
30	1.5	3.4
40	1.4	5.1
50	1.5	4.4

The distribution over most of the panel is apparently not adversely affected by an increase in the forward current density. There is more build-up at the extreme edges (micrometer measurement) at the higher forward current density than at 30 asd and below. Figure 7 shows the coulometric ratios at the current densities tried.

In other tests in the dilute (100 g/l CrO_3) and the standard (250 g/l CrO_3) plating baths, forward current densities considerably higher were tried. A summary of the results follows:

75 asd forward current	Std. soln.*	1.2 coulometric	1.8 micro.
100 asd forward current	Dilute soln.**	1.0	1.4
100 asd No PR	Dilute soln.**	1.2	1.5
100 asd No PR	Std. soln.	1.3	5.0
100 asd forward current	Std. soln.	1.4	2.0
150 asd forward current	Std. soln.	1.3	2.8
150 asd forward current	Std. soln.*	1.2	1.9
150 asd No PR	Std. soln.*	1.3	1.9

*Plus 20 g/l NaOH **Plus NaCl

It appears that edge build-up in the standard bath is greatly reduced by PR but evaluating the effectiveness of PR at the very high forward current densities is difficult because of nodule and dendrite formation at the edges. These growths protect the edge area from excessive build-up during plating and then are removed during rinsing or are too fragile to contribute to the edge thickness measurement. So a panel that actually had considerable build-up at the edges in these forms may appear better in the measured thickness than those with actually better distribution. However, later tests on screw threads indicated that these high current densities would be of limited use on irregularly shaped work.

Reverse Current Level Tests

Several panels were plated to find the most effective reverse current level and forward current level combination for use in PR plating. Various current densities were tried from one-half the forward current value to over five times the forward current. The ratios for low reverse current in the 500 g/l CrO_3 bath averaged 1.2 for the longer cycles and 1.7 for the shorter cycles. Ratios for deposits made during these tests without PR averaged 1.9. Some of the conditions including low reverse current that gave the better deposit ratios were as follows:

180-36 sec.	10-1 eff. cyc.	+30-15 asd	light matte	0.7 ratio
180-30	12-1	+30-15 asd	satin	1.2
180-24	15-1	+30-15 asd	satin	1.3
60-12	10-1	+30-15 asd	satin	1.0
22.5-2	22-1	+30-15 asd	matte	1.4
22.5-5	10-1	+30-15 asd	dark satin	1.3
10-1/2	40-1	+30-15 asd	dark satin	1.4
10-1/4	80-1	+40-20 asd	dark satin	1.4
10-1	20-1	+40-20 asd	matte	1.4

Several tests at 20 asd and at low reverse conditions resulted in bare areas on the specimen even at effective cycles as high as 40-1. The same conditions gave continuous deposits at higher current densities.

The average ratio was 1.4 for equal reverse current in the 500 g/l CrO_3 bath. Two-thirds of the deposits did not completely cover the panel at the low effective cycles used, 15-1 or less. About one-half of the panels plated at similar conditions but with low reverse current did not completely cover.

Some of the conditions giving good results with equal reverse current were:

120-12 sec.	10-1 eff. cyc.	+30-30 asd	satin surface	1.3 ratio
60-4	15-1	+30-30 asd	light matte	1.3
180-18	10-1	+30-30 asd	satin	1.4
120-3	40-1	+30-30 asd	light matte	1.3
60-3	20-1	+30-30 asd	dark matte	1.4
40-2	20-1	+50-50 asd	matte	1.3
60-3	20-1	+50-50 asd	satin	1.3
120-3	40-1	+50-50 asd	satin	1.3
180-9	20-1	+50-50 asd	satin	1.1

The tests shown above that are below the line were run in a 100 g/l CrO_3 bath. The average ratio was 1.3 for the tests in this dilute bath. PR with equal reverse gives distribution ratios equivalent to those obtained by low reverse current under the proper plating conditions and is more efficient as less resistance is needed in the circuit for controlling the current during the reverse period.

The high current reverse tests, of course, used shorter reverse times to obtain the desired effective cycle. They were run in the 500 g/l CrO_3 bath. The average thickness ratio was 2.0 for these tests. Figure 8 shows the average thickness ratios for all fully covered panels at three levels of reverse current. Some of the more favorable conditions for use with a high reverse current are shown below:

180-9 sec.	9-1 eff. cyc.	+30-68 asd	satin surface	1.1 ratio
180-2	12-1	+30-50 asd	satin surface	1.3
60-1/3	40-1	+30-50 asd	light matte	1.4
60-1	11-1	+30-165 asd	matte	1.2
120-2	12-1	+30-165 asd	light matte	1.3
120-2	20-1	+50-150 asd	satin	1.4

Good distribution can be obtained with high reverse current but the higher average ratio for the test indicates that the conditions required are somewhat more critical than at lower reverse current levels. These higher reverse current conditions can be used for plating PR in a chromium bath without adding resistance to the circuit and are thus the most efficient (electrically) and easiest to set up for. The reverse time would have to be adjusted according to the reverse current obtained on a particular piece of work being plated. Thereby the effective cycle giving the best distribution of the deposit could be obtained. It may also be noted that the effective cycles were slightly lower for the high reverse current resulting in a lower average plating rate efficiency requiring a little longer time to plate a given thickness of chromium.

Additives in the Chromium Bath used for PR Plating

Oxalic Acid

A series of tests were made after adding a number of chemicals not normally a part of the composition of a chromium bath. The first tried was oxalic acid in amounts from 5 to 20 g/l added to the 500 g/l CrO_3 bath. The main effect of the oxalic acid addition was an improvement in the appearance of the PR deposits. The chromium was lighter and more reflective.

Oxalic acid forms trivalent chromium in the normal hexavalent solution in roughly the proportion of one part Cr^{+3} for two parts oxalic acid. The Cr^{+3} thus formed is most likely the effective agent in improving PR deposits. However, the amount of hexavalent chromium in the bath is proportionately reduced. Increasing oxalic acid concentration did not result in improvement beyond the normal limits of maximum Cr^{+3} concentration in a bath before standard chromium deposits start to deteriorate as to brightness and hardness.

Additions of oxalic acid had to be made periodically to maintain the Cr^{+3} as the Cr^{+3} became oxidized at the anode. Controlling anode size could avoid additions. The results would indicate improved appearance when using PR for plating chromium on inside surfaces where anode area is restricted and Cr^{+3} concentrations are usually higher.

Figure 9 shows the effect of oxalic addition to the 500 g/l CrO_3 bath. Average thickness ratios for the tests run in the 500 g/l CrO_3 bath are: 1.44 for 5 g/l oxalic acid, 1.37 for 10 g/l and 1.25 for 20 g/l. Three runs without PR with 10 g/l oxalic acid had an average ratio of 1.5. Some of the conditions selected from the test data resulting in good chromium distribution are shown below:

5 g/l Oxalic Acid

120-1/2	60-1 eff. cyc.	+40-60 asd	satin surface	1.3 ratio
120-1	40-1	+50-150 asd	matte	1.1
120-2	60-1	+30-30 asd	satin	1.3
120-6	40-1	+30-15 asd	satin	1.2

10 g/l Oxalic Acid

120-3	40-1 eff. cyc.	+30-30 asd	satin surface	1.3 ratio
120-2	60-1	+30-30 asd	satin	1.2
60-1	60-1	+30-30 asd	satin	1.2
60-1	60-1	+40-120 asd	satin	1.1
45-1	15-1	+40-120 asd	satin	1.3
120-2	20-1	+40-120 asd	satin	1.3
60-1/2	20-1	+50-100 asd	satin	1.2
120-6	20-1	+30-15 asd	satin	1.2

20 g/l Oxalic Acid

120-2	20-1	+30-90 asd	satin	1.3
60-1	20-1	+30-90 asd	satin	1.2
60-1	20-1	+30-90 asd	satin	1.1
30-4	15-1	+50-25 asd	satin	1.3
40-4	20-1	+50-25 asd	satin	1.1

Oxalic acid was also added to the dilute 100 g/l CrO_3 bath. The average ratio was 1.2 for three runs with PR but without oxalic in this bath. After the addition of 10 g/l oxalic acid the average ratio was 1.1 for eleven runs. The first two runs in the bath had a ratio of less than one which is one of the few times during this investigation that a reversal of the normal distribution characteristics of chromium deposits was achieved. Data on a few runs with 10 g/l oxalic acid in the dilute bath that had the best ratios are shown below:

120-6 sec.	40-1 eff. cyc.	+50-25 asd	satin surface	0.9 ratio
40-4	20-1	+50-25 asd	light matte	1.1
120-12	20-1	+30-15 asd	light matte	1.1
120-6	40-1	+50-25 asd	satin	1.1
120-6	40-1	+50-25 asd	semi-brt.	1.0

Under the above conditions a rather mild (slightly sacrificial) PR cycle resulted in excellent distribution. The principle effect of the addition of oxalic acid to a chromium bath used with PR seems to be in brightening and smoothing the generally dull PR deposit but did result in slightly better deposit distribution than any of the other experiments.

Boric Acid

Boric acid was also tried as an additive at several concentrations to the 100 and 250 g/l CrO_3 baths. Although the appearance of the deposits was improved, little general improvement in deposit distribution was noted. Average thickness ratios for PR deposits made with various boric acid concentrations in the dilute bath were: 1.4 for 5 g/l, 1.3 for 10 g/l, 1.35 for 20 g/l, 1.3 for 50 g/l, and 1.9 for 100 g/l. These data may be compared with those in the table under bath concentration. In the standard 250 g/l CrO_3 bath the ratio was 1.35 for 50 g/l H_3BO_3. The standard bath without addition of PR yielded a thickness ratio of 1.8 at 30 asd and 1.4 at 50 asd. Additions of boric acid without PR gave ratios of 1.3 and 1.5 for dilute and standard baths respectively. Figures 10 and 11 show effects of boric acid additions.

Sodium Hydroxide

Sodium hydroxide was added to the 250 g/l CrO_3 bath resulting in somewhat improved deposit distribution with and without PR. Concentration of this additive was limited to less than 50 g/l because of burning and flaking of the chromium on the high current edges of the panels. The current density range of deposition was apparently greatly reduced. Appearance of the deposit was not adversely affected by adding up to 25 g/l NaOH to the bath. Average deposition distribution ratios for NaOH additions to the 250 g/l CrO_3 bath are: 1.5 for 10 g/l and 1.4 for 25 g/l. The effect of NaOH additions on thickness ratios is shown in Figures 12 and 13. The addition of 25 g/l NaOH resulted in non-plated edges in the dilute bath but did not have this effect in the standard bath. That the allowable amount of added NaOH is proportional to the CrO_3 concentration is demonstrated by the non-plated edges obtained when 25 g/l NaOH was added to a dilute bath. The edge effect was thought to be related to passivation of the steel panel. Some of the conditions found to give good results when using NaOH as an additive in the 250 g/l CrO_3 bath were:

60-6 sec.	20-1 eff. cyc.	+50-25 asd	satin surface	1.2 ratio
120-6	40-1	+50-25 asd	satin surface	1.2
60-6	40-1	+50-25 asd	satin surface	1.2
No PR	-	+30 asd	bright	1.2
No PR	-	+50 asd	bright	1.3

In the 100 g/l CrO_3 bath the average thickness ratio was 1.2 with 10 g/l NaOH added using PR and 1.3 without PR. No PR and no NaOH gave 1.4.

Some of the plating conditions tried which resulted in the best thickness ratios are shown below:

120-6 sec.	20-1 eff. cyc.	+50-50 asd	light matte surface	1.1 ratio
60-3	20-1	+50-50 asd	matte	1.1
180-4 1/2	40-1	+50-50 asd	bright satin	1.2
120-3	40-1	+50-50 asd	satin	1.2
40-1	40-1	+50-50 asd	matte	1.2
60-1/2	40-1	+50-50 asd	light matte	1.2
60-6	20-1	+50-25 asd	matte	1.2
180-9	20-1	+50-50 asd	satin	1.2

Consistently good results were obtained using PR cycles ranging from 40-1 sec to 180-12 sec. Several runs without PR had an average ratio of 1.3 at 30 and 50 asd. Deposit distribution ratio was 1.4 for several runs in the dilute bath with no additive or PR. The effects of NaOH addition on the deposit is shown in Figure 14.

Sodium Chloride

Another sodium compound, sodium chloride, was tried as a leveling additive in the chromium bath. It was used in the dilute (100 g/l CrO_3) bath in amounts from 5 to 30 g/l. At the highest concentration it caused burnt or bare edges on the panel in four out of five tests. Most of the tests were run at 50 asd and with three runs at 100 asd. A summary of the NaCl concentrations and the resulting thickness distribution ratios is shown in Figure 13 and below:

5 g/l NaCl	1.3 ave. ratio	1.2 median ratio
10	1.2	1.2
15	1.2	1.1
20	1.0	-
30	1.2	1.2

No deposition or burning occurred on the extreme edges of the panel even without PR. Some chlorine gas was released at the anode and the effect of an addition of NaCl decreased after a few runs. Such conditions would probably make the addition of NaCl to the chromium bath of limited use.

Sodium Carbonate

A few tests were made of the benefits of adding sodium carbonate for improving the distribution of chromium. The results were not as good as for some of the other additives tried. For tests using 10 to 20 g/l of $NaCO_3$ in the dilute CrO_3 bath the average and median ratio was 1.3.

Summary of Additives

An overall comparison of the coulometric (C) and micrometer (M) thickness data obtained using additives in the CrO_3 bath on the basis of current densities used is given below:

		Deposit Thickness Ratios	
		of C1.5 and M3.5 or less	C1.3 and M3.0 or less
Oxalic acid at	30 asd	70%	30%
(conc. & dil. bath)	40	83	50
	50	74	53
Boric acid at	30	87.5	50
(dil. & std. bath)	50	100	80
Sodium hydroxide at	30	55	29
(dil. & std. bath)	50	93	79
Sodium chloride at	50	100	82
(dil. bath)	100	100	100
Sodium carbonate	50	89	56

Bath Temperature and Deposit Appearance

A series of runs were made in the dilute (100 g/l CrO_3) bath at 30 asd with all conditions including the PR cycle the same except that the temperature was varied from 35 to 90°C. The temperature resulting in the best appearance - smoothest and brightest deposit - was 65° with 60 and 70° nearly as good. This temperature is only about 10-15° higher than normally used in chromium plating and it would seem practical to make PR deposits at this temperature for the benefit of improved appearance.

The 65° temperature did not give the best distribution of the deposit at the PR cycle used (120-3 sec) so a series of PR cycles was tried at the same conditions to find a combination that would result in improved distribution as well as improved appearance. Several PR cycles at 20 asd did not result in the desired low thickness ratios but at 50 asd most of the PR cycles tried resulted in ratios of 1.3 or better.

Several of the better combinations at 65°C giving good surface appearance are detailed below:

High Reverse Current

120-1-1/2 sec.	40-1 eff. cyc.	+50-100 asd	semi-brt. surface	1.3 ratio
120-3	20-1	+50-100 asd	bright satin	1.1
120-4	15-1	+50-100 asd	satin	1.3

Low Reverse

120-12	20-1	+50-25 asd	satin	1.1
120-16	15-1	+50-25 asd	satin	1.3
120-24	10-1	+50-25 asd	satin	1.3
120-6	40-1	+50-25 asd	bright satin	1.2
60-3	40-1	+50-25 asd	satin	1.3
20-2	20-1	+50-25 asd	light matte	1.2

Equal Reverse

20-1/2	40-1	50-50 asd	matte	1.2
40-1	40-1	50-50 asd	light matte	1.3
60-1-1/2	40-1	50-50 asd	light matte	1.2
180-4-1/2	40-1	50-50 asd	bright satin	1.1
60-3	20-1	50-50 asd	satin	1.1
40-2	20-1	50-50 asd	light matte	1.3

The average deposit distribution ratio for this series at 50 asd was 1.4. The average ratio of 1.5 was obtained for similar runs at 55°C.

Cross-Section of PR Deposits

The hardness of a number of deposits was made on the cross-section using a microhardness machine with a Knoop indenter and a 200 g load. The deposits were sectioned and mounted in bakelite to make the measurements. An indication of the edge build-up or thickness ratio was also obtained from photographs of the cross-sections at the edge and edge + 1.5 cm. Some of these pictures are included at the end of this report.

PR deposits and deposits plated using bath additives without PR showed some decrease in hardness over standard bright chromium deposits but most retained considerable hardness as shown in the data below. These data indicate that PR deposits would be hard enough for most applications of chromium.

			(edge-edge + 1.5 cm)
No PR dilute bath	50 asd	1077 KHN_{200}	2.5 ratio
No PR dilute bath	30 asd	1006	2.3
No PR dilute bath	30 asd	983	1.7
No PR dilute bath	20 asd	934	2.7
No PR 10 g/l oxalic acid	30 asd	777	3.4
No PR 20 g/l boric acid	30 asd	797	1.6
No PR 30 g/l boric acid	30 asd	999	1.6
No PR 25 g/l NaOH	30 asd	947	1.3
No PR 25 g/l NaOH	50 asd	1037	1.6
60-1 sec. dilute bath + boric	+30-90 asd	781	3.1
120-2 std. bath + NaOH	+30-90 asd	693	2.4
60-1-1/2 dilute bath	+30-90 asd	469	6.0
60-1 dilute bath	+30-90 asd	824	2.4
120-1 dilute bath	+30-90 asd	723	2.1
60-3 dilute bath	+30-30 asd	812	2.2
120-3 oxalic acid addn.	+30-30 asd	589	6.0
120-6 oxalic acid addn.	30-15 asd	700	4.7
120-6 dilute + oxalic	30-15 asd	969	1.6
120-6 boric acid addn.	30-15 asd	638	1.6
120-6 NaOH addn.	30-15 asd	973	2.0
120-6 dilute bath	30-15 asd	983	1.3
60-6 dilute bath	30-15 asd	743	2.0
120-6 dilute bath	30-15 asd	664	2.6
40-4 dilute bath	30-15 asd	756	1.8

A simplified comparison is given by averages of the hardness data:

No PR - 1000 KHN_{200}
No PR plus addn. - 911
PR - high reverse - 698
PR - equal reverse - 701
PR - low reverse - 803

From the standpoint of hardness it appears that a low PR reverse current is preferable and the thickness ratios are also good.

Combination of Favorable Conditions

There were several plating conditions or bath modifications that seemed to improve chromium distribution or appearance. A few tests were made combining several of these favorable conditions in hopes that they were additive and thus produce the best possible chromium deposit. The tests were made in a dilute (100 g/l CrO_3) bath with the addition of 10 g/l NaOH and used reverse current equal to or lower than the forward current at 50 asd and 65°C. The deposits were all satin or better in appearance and had deposit thickness ratios of 1.3 or better but only one had a deposit ratio of 1.0 which was obtained with a PR cycle of 120-6 sec, effective cycle of 20-1 and equal reverse current. A control deposit plated without PR had a ratio of 1.3 but the extreme edge build-up was slightly higher than for any of the runs with PR.

Screw Threads Plated with Best PR Conditions

A number of 1/4"-20 screw threads were plated with chromium in the dilute (100 g/l CrO_3) bath and in the standard (250 g/l CrO_3) bath. Best current and PR conditions for obtaining uniform deposit distribution on a panel were not necessarily best for threads. Reverse currents higher than about 65 asd resulted in poorer distribution than without PR. Cycles that were too sacrificial, larger proportion of reverse time and current to forward time and current, resulted in no deposit in the bottom of the grooves of the threads. A forward current density of 50 asd with PR worked well on panels but currents of 30 or 20 asd worked better on threads. It may have been that gasing in the thread grooves increased resistance of the solution in this critical area more or less in proportion to the current density. With virtually no gas involved during reverse, the benefits or PR were reduced. Only additions of NaOH were tried during these runs and it improved distribution without the use of PR in the standard bath and gave some advantage with PR in the dilute bath.

Despite the factors working against good deposit distribution, thickness ratios between the top and bottom of the threads in the 250 g/l bath were reduced from a low of 2.7 without PR to 1.3 with PR at similar bath conditions. About the same or poorer distribution is obtained from an electroless nickel bath without special agitation of the solution.

Some of the conditions giving relatively good results on the threads were:

Dilute + NaOH	55°C	120-3 sec.	20-1 eff. cyc.	+50-100 asd	2.3 ratio
Dilute + NaOH	55°C	120-3	20-1	+30-60	2.
Dilute + NaOH	55°C	120-6	40-1	+30-15	2.
Dilute + NaOH	55°C	120-6	40-1	+20-10	1.5
Dilute + NaOH	55°C	120-9	25-1	+30-15	2.5
Dilute + NaOH	65°C	120-12	20-1	+50-25	2.5
Std. Bath	65°C	120-3	20-1	+30-60	1.3
Std. Bath	65°C	120-6	20-1	+30-30	2.
Std. Bath	65°C	120-5	20-1	+50-65	2.
Std. + NaOH	65°C	120-3	20-1	+30-60	1.3
Std. + NaOH	65°C	No PR	-	+30	3.
Std. + NaOH	65°C	No PR	-	+50	5.

The average of all seven deposits without PR was 3.5.

The results of plating the threads in comparison to the results on the panels indicate that it may be necessary to adjust the PR cycle to the type of contour being chromium plated to obtain the most uniform deposit distribution. Also the standard (250 g/l CrO_3) chromium bath (average ratio 3.3) seems to work as well or better than the dilute (100 g/l CrO_3) bath (average ratio 3.7) when plating threads. This is not necessarily true for panels. Photographs of several cross-sections of the threads are included as Figure 15.

SUMMARY OF PR CHROMIUM PLATING TESTS

Some of the factors involved in PR chromium plating which were made evident during these tests are listed below:

1. Reverse current efficiency is about six times plating efficiency.

2. Chromium is dissolved from the cathode in the hexavalent state.

3. Periodic reversal of the current for periods of less than 50 seconds has no detectable adverse effect on the continuity of the deposit at normal current densities.

4. The reverse portion of the PR cycle produces 2-3 times the plating current at plating voltage.

5. The PR cycle that gave the lowest ratios was 120-6 sec. with reverse current one-half plating current in the dilute bath plus oxalic acid.

6. Short sacrificial PR cycles yielded dark matte deposits but usually better uniformity of thickness under most bath conditions.

7. Smoothness and brightness of PR deposits may be improved by plating at end of run for a few minutes without reversing.

8. PR cycles longer than three minutes cause sludging of the anode especially in concentrated CrO_3 solutions because of the amount of reverse current necessary for thickness ratio improvement.

9. The higher current density regularly used (50 asd) gave best distribution on flat panels with and without PR.

10. Reverse currents that were higher than 4 times forward current with effective cycles of 15-1 or less tended to dissolve so much chromium from low current density areas that poorer distribution or even bare areas resulted.

11. Addition of up to 25 g/l of oxalic acid, boric acid or sodium hydroxide improves appearance of PR deposits, distribution of standard deposits and PR deposits with oxalic acid.

12. Rainbow colored oxides form on steel areas below plating current density during PR plating in CrO_3 bath.

13. The range of hardness of PR deposits is less than that of standard bright chromium.

14. Appearance and distribution of PR chromium improved by increasing the level or Cr^{+3} in CrO_3 bath, e.g., by the addition of oxalic acid to the bath.

15. Conditions giving improved deposit distribution with PR chromium not necessarily additive.

16. Threads may be plated with PR chromium with a thickness ratio of only 1.3.

EQUIPMENT FOR PULSED CURRENT TESTS

A current supply that produced millisecond pulses of dc current was obtained from Rapid Electric Company. The unit had five controls, two of which were timers with which pulse-time off could be controlled within

0-100 ms. A rheostat adjusted the pulse voltage, another adjusted the percentage of the pulse voltage desired for the base current or fraction of the pulse voltage to be applied to the work between pulses. A third rheostat limited the pulse current provided it was set for a current less than that delivered by the voltage setting. The shape of some pulse wave forms are shown in Figure 16. A Tektronix type 513A oscilloscope was used to monitor the wave form of the pulse. This could be connected to either the voltage or current output of the pulser. The remainder of the plating set-up was the same as for PR plating with heavy, well spaced leads used to supply current to the work for minimizing induction.

RESULTS OF PULSED CURRENT TESTS

Pulse time and level tests

Initial tests in the 250 g/l CrO_3 bath indicated that at both 30 and and 45 amperes per square decimeter and at 65°C there was some improvement in the distribution of the chromium deposit on the 3 x 3 flat polished steel panels at certain pulse conditions. It was also discovered that the base setting of current between pulses must be above 20 percent of the pulse voltage to obtain at least a satin surface on the chromium deposit. A test, described later, indicated that the uncertainty of deposit distribution reproducibility was about 18%. It was found at this time that a reverse current of 1 to 3 minutes before plating did not give adequate adhesion at the edges when plating with a pulse of more than 30 asd. A forward current below plating level for one minute after a one minute reverse solved the adhesion problem.

Pulse time variables at 55 and 65°C

A series of tests were made with varying pulse-on and pulse-off times at 65°C and 45 asd in the 250 g/l bath using a 25% base setting between pulses. Current density as measured by the meter is an average of pulse and base current. Pulse current increased as pulse time and base level decreased and as time between pulses increased. Two pulse conditions, 10 ms. on - 30 ms. off and 2 ms. on - 2 ms. off gave edge to center thickness ratios of 1.7 and 1.9 respectively as measured with a micrometer. Two tests without pulsing at the same conditions yielded thickness ratios of 2.3. At 65°C both of the above low ratio deposits had a matte surface. Poorest ratio was 2.4 at 50-10 ms. At 55°C and 45 asd the best ratios of 2.0 were obtained at 30-30 and 10-10 ms., the poorest of 2.4 at 3-2 and 5-5 ms. The deposits were dull.

Base voltage

In addition to a base voltage of 35% of pulse voltage for between-pulse current setting, several other base levels were tried. The literature (listed in the bibliography at the end of this report) recommended one third pulse voltage for the base setting for chromium plating. A number of tests were made at 33% of pulse voltage and at several pulse-times. A number of tests were also run with the pulse-time at 7 asd on and 21 asd off with only the base setting varied. All tests were run in the 250 g/l CrO_3 bath at 55°C and 45 asd. At 75% base setting the best thickness ratio of 1.4 and plating efficiency of 14.4% was obtained. A later confirmatory test gave a ratio of 1.9 and efficiency of 14.0%. At 60% base setting the ratio was 1.7 and plating efficiency 13.0%. All three deposits were bright. This test indicates that the relatively high base settings of 60 to 75% yielded the best results at least at the plating condition used. A tabulation of the data is shown below.

Base Setting % pulse	Surface	Efficiency	Cr Thickness Ratio edge - center
33%	semi-bright	9.8%	2.3
33	semi-bright	6.9	2.0
40	near-bright	8.1	1.8
40	bright	9.7	2.0
45	bright	9.5	2.4
50	bright	12.3	2.4
50	semi-bright	11.9	2.4
60	bright	13.0	1.7
75	bright	14.4	1.4
75	bright	14.0	1.9

Figure 17 is a plot of plating efficiency versus base voltage settings.

Hardness tests of PC deposits

Cross-sections were made of several deposits plated with a range of pulse ratios and at 65°C/45 asd in the 250 g/l CrO_3 bath. There was little relation between the surface appearance of the deposits and the hardness. Two bright deposits had a hardness of about 600 and 700 KHN_{200} respectively and two matte deposits measured about 875 KHN_{200}. On the average the longer cycles of 30-30 and 50-50 ms. resulted in the harder deposits. Shorter cycles of 2-2, 10-10, and 10-30 ms. gave the softest deposits with a low of 357 KHN_{200}.

Individual measurements are shown below:

Pulse (ms)	Temp/CD	Hardness (KHN_{200})
50-50	65/60	835
50-50	65/45	692
50-50	65/45	876
30-30	65/45	875
10-10	65/45	595
10-10	65/45	823
10-30	65/45	357
2-2	65/45	449

Pulse ratio tests

Further exploration of pulse on-pulse off time ratios were made at 55°C/45 asd at base voltage settings of 33 and 50% of pulse voltage. Confirmation of the results of improved surface appearance at higher base settings was obtained. Ten of the 24 deposits made at 33% base voltage setting were matte while none of those made at 50% were matte. The poorest surface at the latter setting was a bright satin and most were semi-bright or bright. Twenty-one deposits were made at 50% base setting with an average thickness ratio of 2.1 while the average of twenty-four made at 33% was 2.4 although there were more low (1.8 or less) thickness ratios at the 33% base setting. For example, a pulse time of 20-60 ms. gave a thickness ratio of 1.6. The best thickness ratio at 50% base setting was 1.7 obtained at 10-5 and 15-15 ms. pulse times. A pulse-time ratio of 1 on to 3 off showed evidence of being preferable. A test at 25% base setting and 10-10, 10-20, 10-30 ms. pulse timing gave thickness ratios of 2.3, 2.2, and 1.7 respectively. While some other pulse times such as 50-50 ms. and 10-5 ms. gave occasional good results pulse-time ratios with pulse times greater or equal to off-time generally gave poorer chromium distribution.

Duplication of low ratio deposits

A number of panels were plated at conditions that duplicated previous tests resulting in better than normal deposit distribution with ratios from 1.8 to 1.4. In most cases only one duplicate was made at each set of conditions and the thickness ratios ran a little higher than the original results in most cases. However, only four of the fourteen deposits had ratios greater than 18% higher than initial deposits and most were less than 12% higher in this test. Pulse times for these low thickness ratios obtained at 55°C/45 asd in the 250 g/l CrO_3 bath ranged from

3-15 through 15-15 and 7-21 to 25-75 ms. A deposit plated with conventional dc at the same conditions had a thickness ratio of 2.5.

Dilute chromium plating bath

The effect of a dilute 100 g/l CrO_3 bath with PC was investigated. As in the PR tests, improvement in deposit distribution was realized in the dilute bath. Also, nearly all the PC deposits were bright. The usual complaint about plating in the dilute CrO_3 bath is the limited bright range.

Nine runs were made in the dilute bath and a range of pulse times and base voltage settings were used. All deposits had a thickness ratio of 1.8 or less with one panel having a nearly uniform thickness with a ratio of 0.9. The ratios of the five best panels averaged 1.3. A deposit plated with standard dc current had a thickness ratio of 1.6. Detailed data for the test are shown below:

Pulse on-off	Base Setting (% pulse)	Temp./CD (°C/asd)	Surface	Efficiency	Cr Thickness Ratio Edge-center
16-49 ms.	33%	55/45	near bright	12.7%	1.4
25-100	33	55/45	bright	11.8	1.3
15-15	50	55/45	light matte	13.7	1.8
10-5	50	55/45	bright	12.2	1.3
3-15	33	55/45	bright	9.7	0.9
50-50	33	55/45	bright	12.4	1.2
7-21	75	55/45	bright	13.4	1.2
10-30	25	65/45	near bright	7.0	1.5
100-0	--	55/45	bright	14.9	1.6

Concentrated chromium plating bath

Eight runs in a 500 g/l CrO_3 bath gave just the opposite results of those obtained in the dilute bath. Most deposits were matte and deposit thickness ratios were high. However, all deposit thickness ratios with pulsed current were considerably lower than the 7.4 ratio obtained using standard dc current. The best ratio obtained in the concentrated bath was 1.6 for a 10-30 ms. pulse at a 25% base setting. The average thickness ratio for deposits made with pulsed current was 3.4. All tests were made at 55°C/45 asd.

Current controlled pulse

Most of the runs made with pulsed current utilized the voltage control to maintain the desired average current and the current control was set to allow the maximum current to flow. A few tests were made with the current control rheostat. No appreciable difference was noted in deposit distribution from the 250 g/l CrO_3 bath at 55°C/45 asd. There was some indication that the appearance might be slightly improved as all the deposits were bright but many deposits from this bath were bright when voltage control of the average current was used.

SUMMARY OF PC CHROMIUM PLATING TESTS

Some of the findings made during the PC tests in the chromium plating bath that might be of more general interest are given below:

1. Distribution of the chromium deposit was improved at some conditions of pulsed current.

2. Pulsed current has no observed deleterious effect on chromium properties, except a possible reduction in hardness, and may improve the brightness at some conditions of deposition and bath composition.

3. Efficiency of deposition was about normal for many pulse-on, pulse-off time ratios but poorer for some than when using standard dc current.

4. Contacts to work should be secure to prevent arcing during high current peak pulses and leads sized and arranged to minimize induction due to high frequency pulsing.

5. Appearance of deposits made with pulsed current is as good or better than those made with regular dc in the standard (250 g/l) or dilute (100 g/l) bath and average much better than those made with PR current.

6. The concentration of CrO_3 in the bath had a pronounced effect on the deposit distribution and appearance when using PC although there was some improvement in uniformity over regular dc current at all concentrations.

7. Improvements in deposit uniformity and appearance were experienced over a wide range of pulse on-off ratios and base voltage settings.

8. There was some indication that certain pulse ratios and base settings were more effective at certain bath concentrations, e.g., the base setting of 33% and pulse ratio of 3-15 ms. that gave a 0.9 thickness ratio in the dilute bath.

9. No brightening of initially dull surfaces was experienced when using pulsed current for chromium plating.

10. Plating rates were changed by changes in the pulse duration and pulse on-off ratios even for the same average current, e.g., in the concentrated bath at 45 asd a 10-5 pulse for 2 hrs. gave 2.3 mils of Cr, a 25-100 pulse 1.6 mils for a respective efficiency of 12.2 and 8.5%.

SUMMARY

Both PR and PC power supplies resulted in somewhat improved distribution of chromium deposits from a wide variety of bath concentrations and current-time conditions. PR current always resulted in deposits that were less bright or smooth on the surface than standard dc. PC improved the appearance of the deposit in baths of low concentration (250 g/l or less). Hardness of the chromium deposits was reduced by both types of power supplies with the greatest reduction by PR although in most cases the hardness still would be effective for wear resistance and greater than that for chromium heated to 600°C. Plating rates varied with the PR or PC time schedule used but no rates were greater than normal for standard dc current. PR seems to be more effective with higher than normal limits of Cr^{+3} (trivalent) in the bath. Although improvements in deposit distribution were shown with PR and PC, the gains are not significant enough to warrant implementation of such power supplies for production chromium plating. Continued development of these processes is necessary, especially, as new electronic power supplies are introduced in the industrial market.

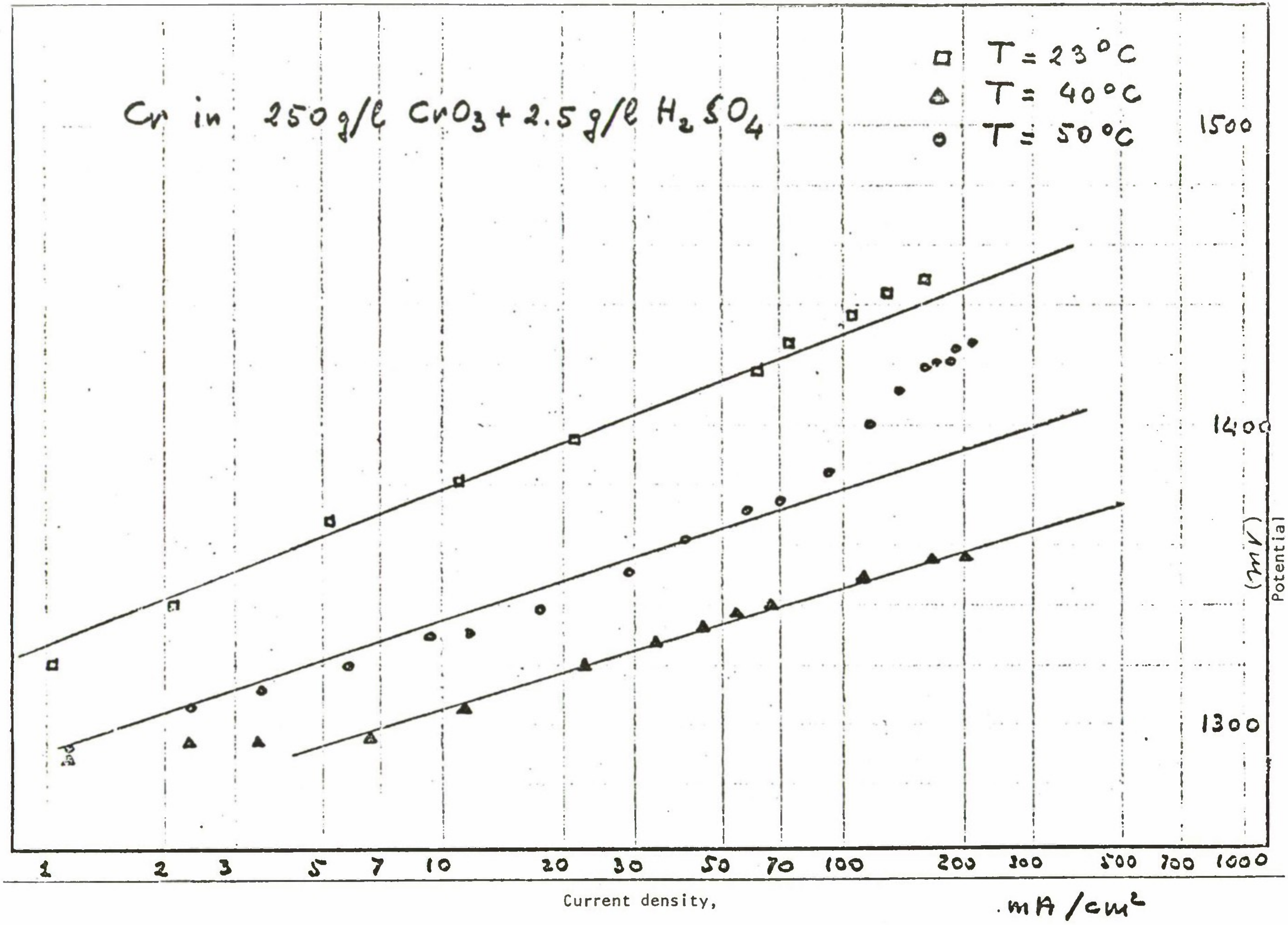


FIGURE 1 Curves for the electrolytic dissolution of chromium by reverse current

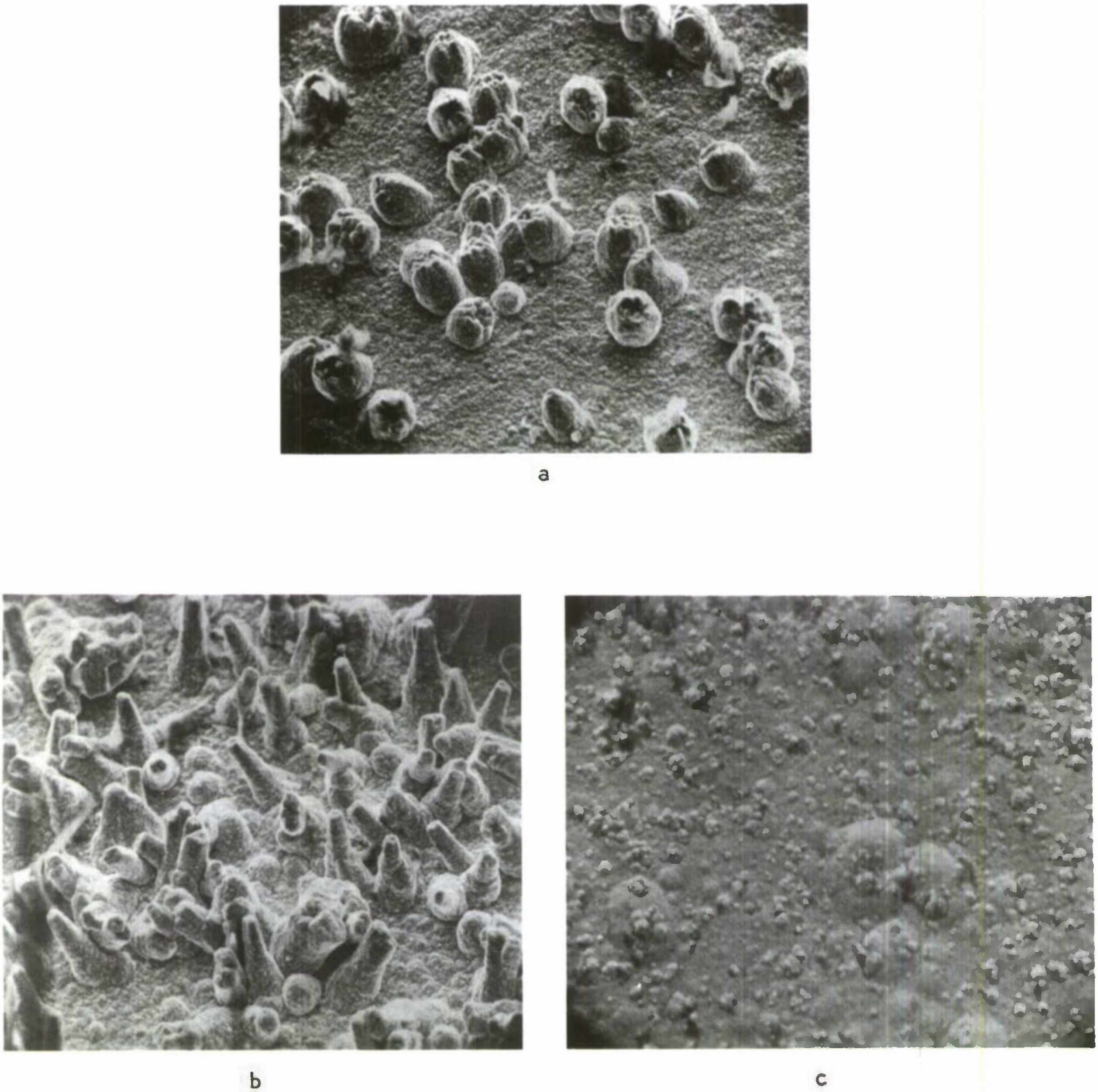


FIGURE 2 SEM photographs of the as-plated chromium surfaces: a) PR 120-3 sec, 50°/20 asd, effective cycle 80-1, satin surface, b) PR 120-1 sec, 50°/30 asd, effective cycle 40-1, matte surface; c) PR 120-1 sec plus 5 g/l oxalic acid in bath, 50°/30 asd, effective cycle 40-1, semi-bright surface. 1100X

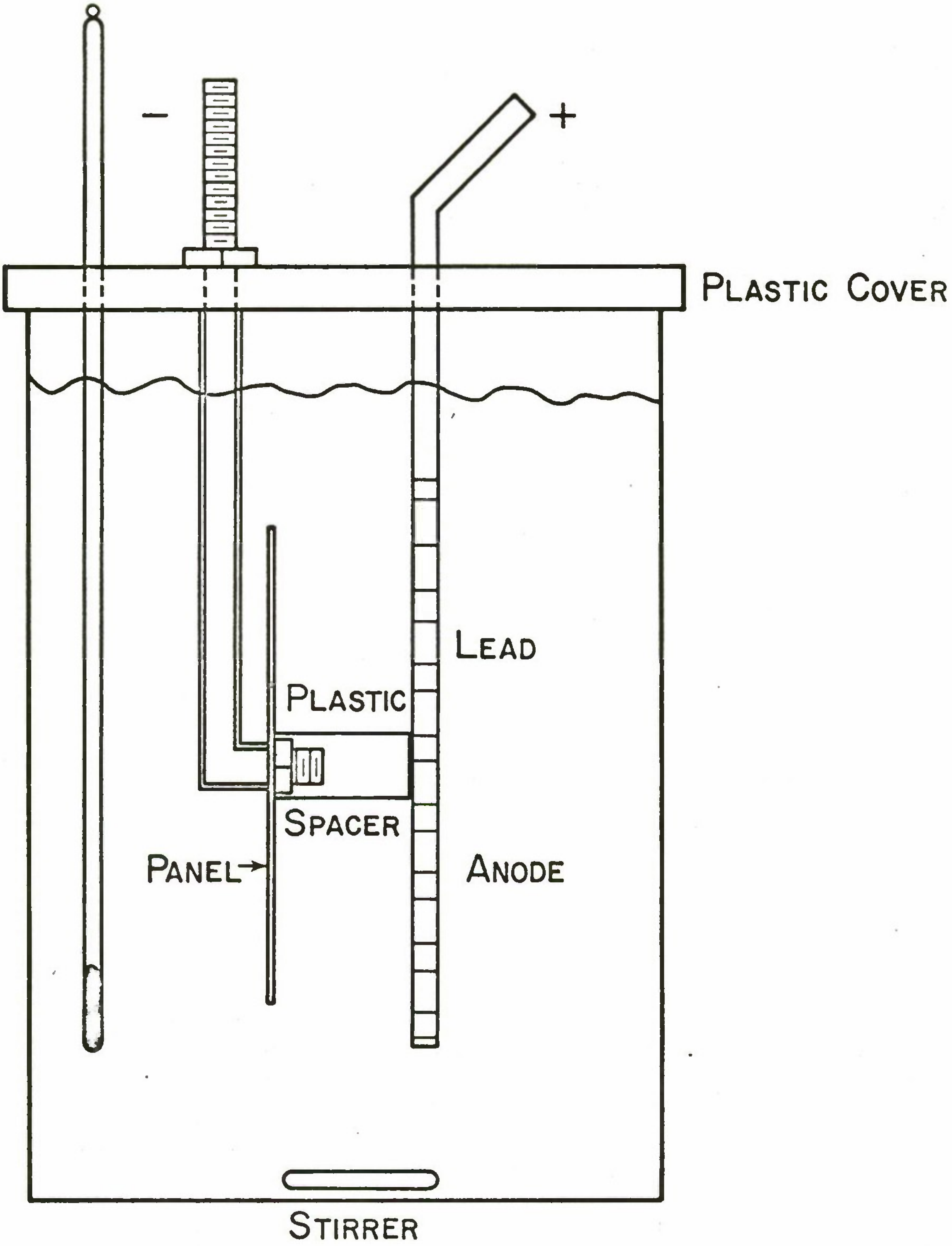


FIGURE 3 Electrode and bath arrangement for experimental PR plating

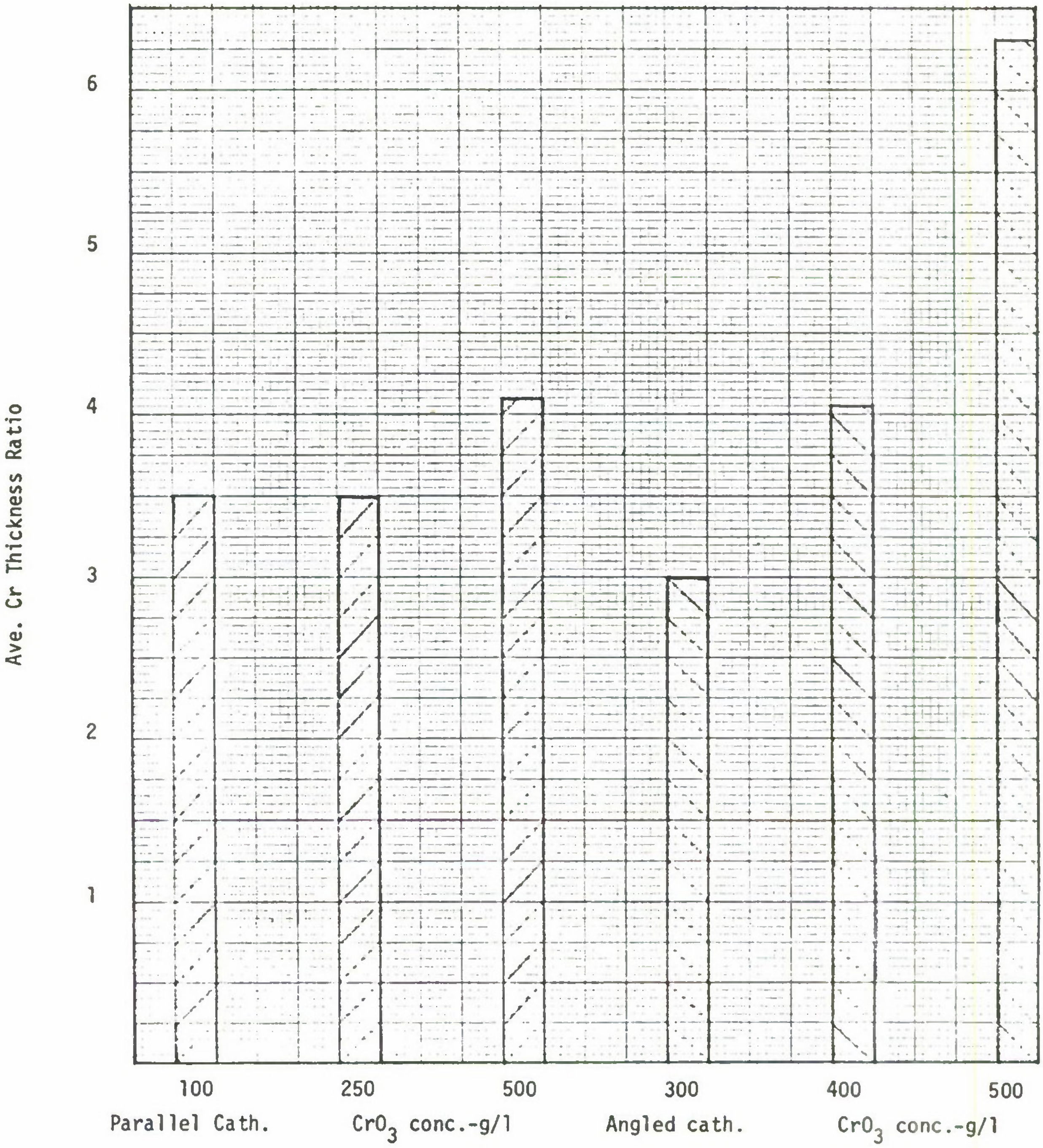


FIGURE 4 CrO_3 bath concentration using angled and parallel cathodes

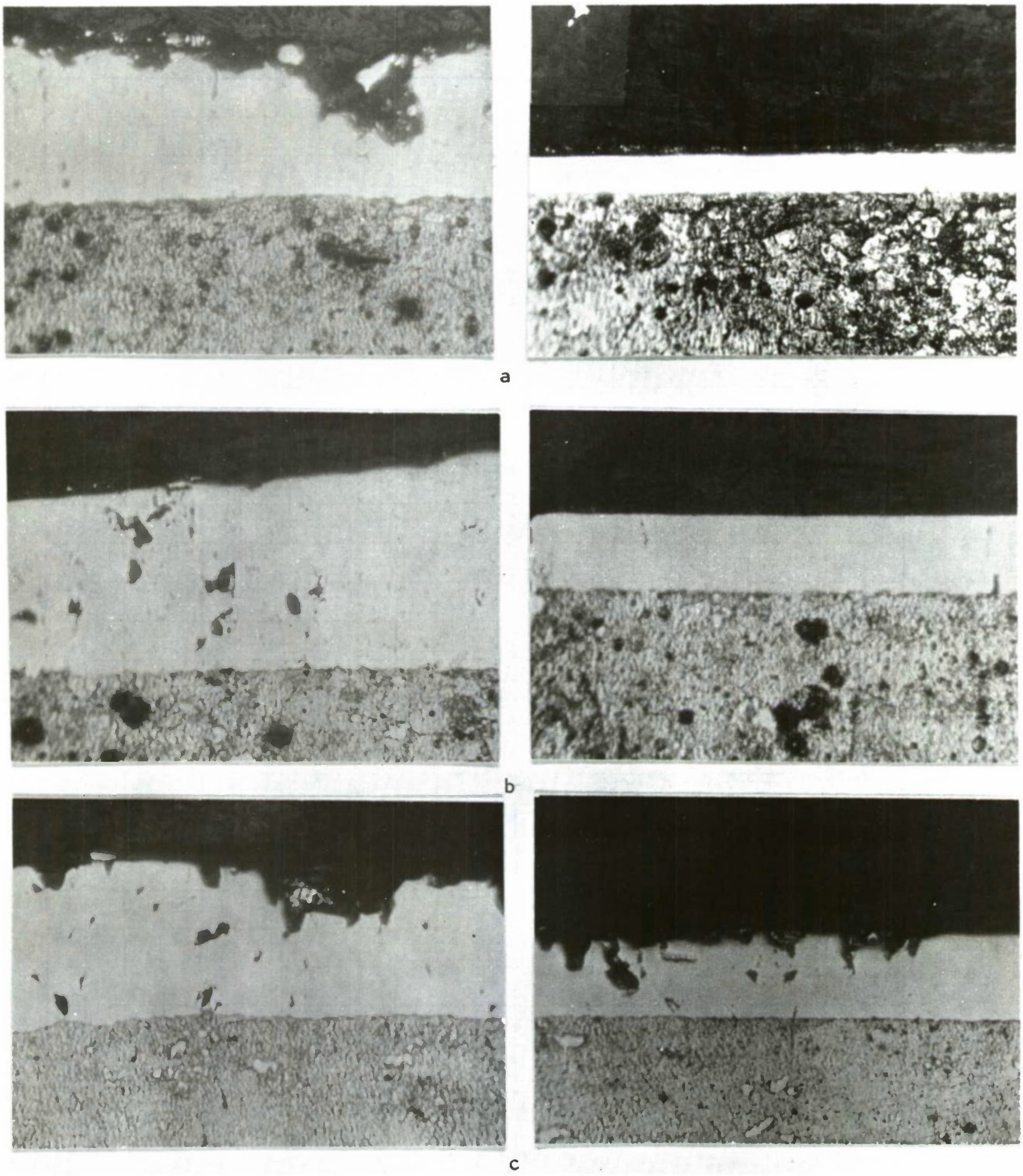


FIGURE 5 Cross-section of deposits without PR at edge (left) and edge + 1.5 cm (right) a) 500, b) 250, c) 100 g/l CrO_3

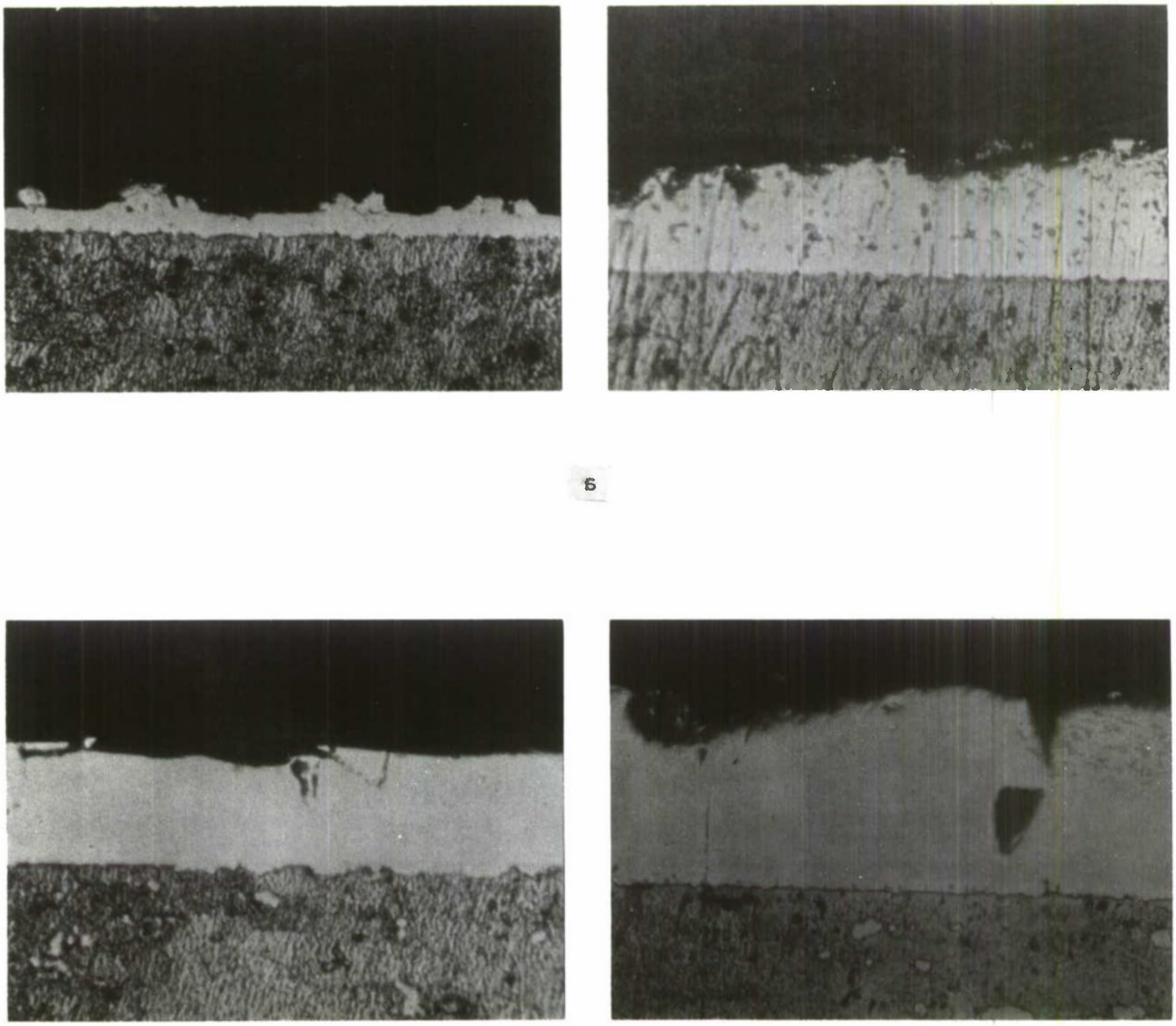


FIGURE 6 (#277-89) Cross-section photomicrographs of PR deposits from 500(a) and 100(b) g/l CrO_3 bath. Edge (left) and edge + 1.5cm (right).

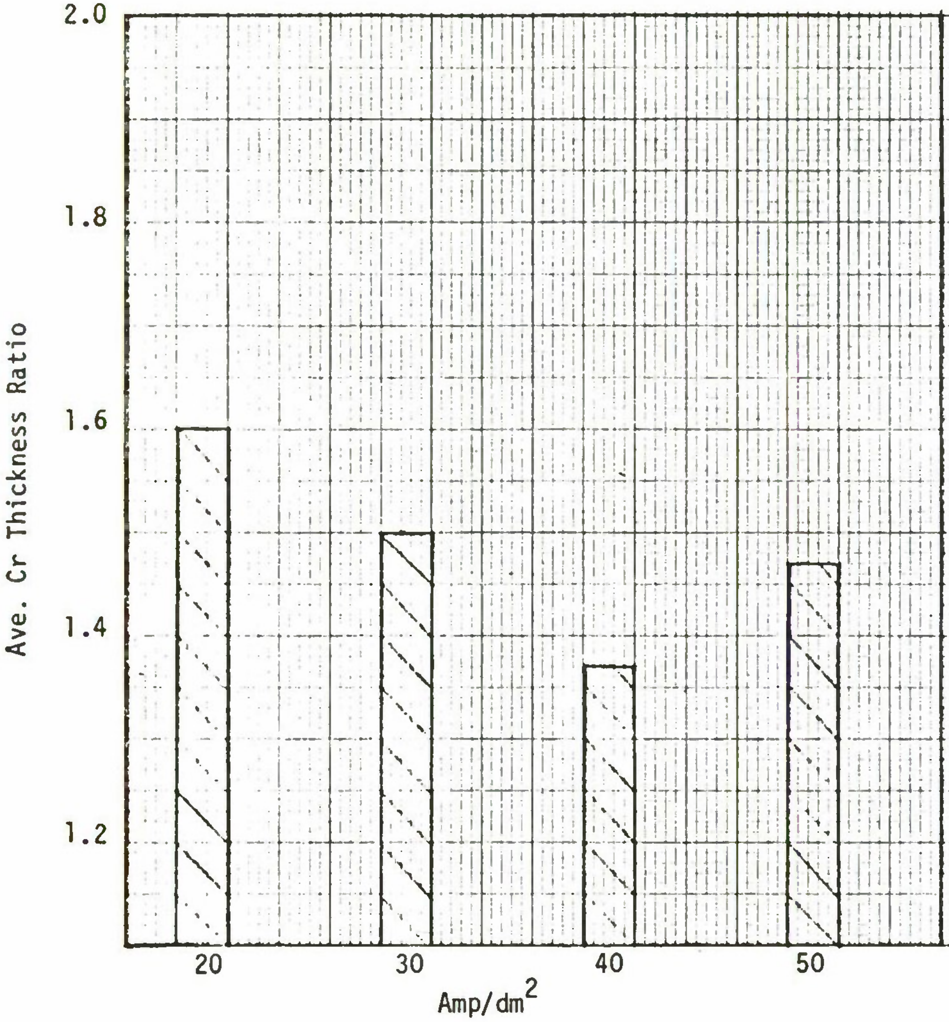


FIGURE 7 Average thickness ratio of PR deposits in 500 g/l CrO_3 bath at various current densities

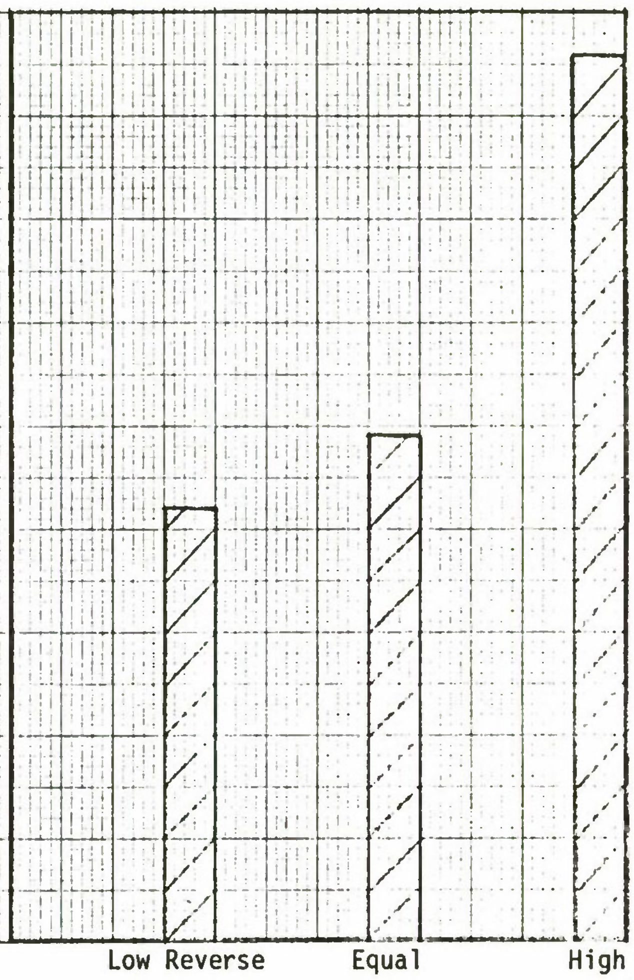


FIGURE 8 Reverse current density level for PR plating in 500 g/l CrO_3 bath

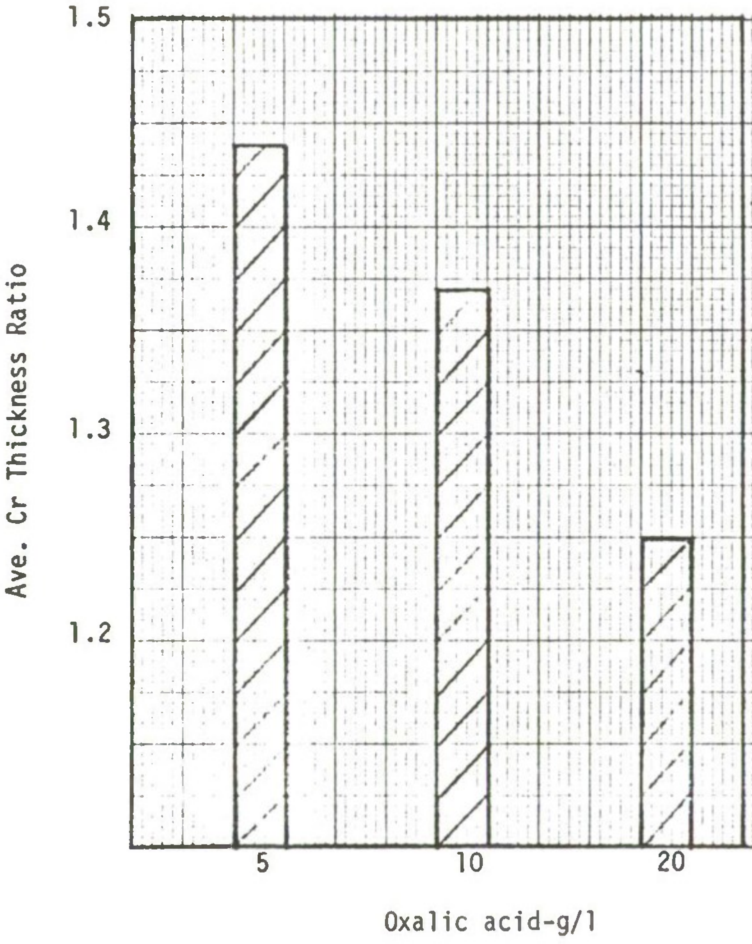


FIGURE 9 Oxalic acid addition to 500 g/1 CrO_3 bath for PR plating

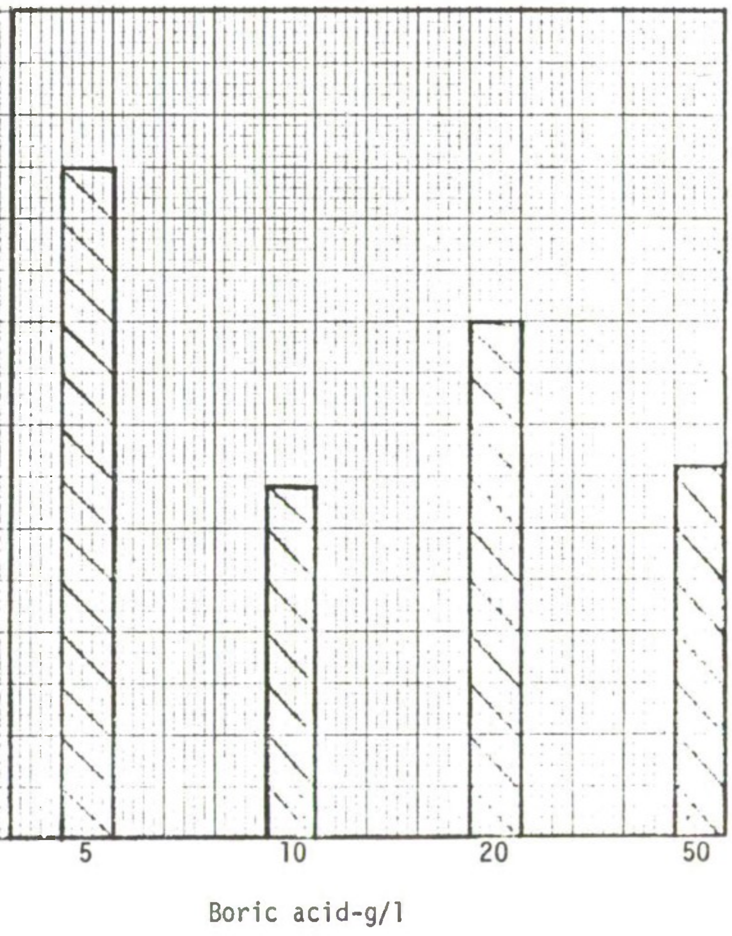


FIGURE 10 Boric acid added to (100 g/1 CrO_3) and (250 g/1 CrO_3 bath)

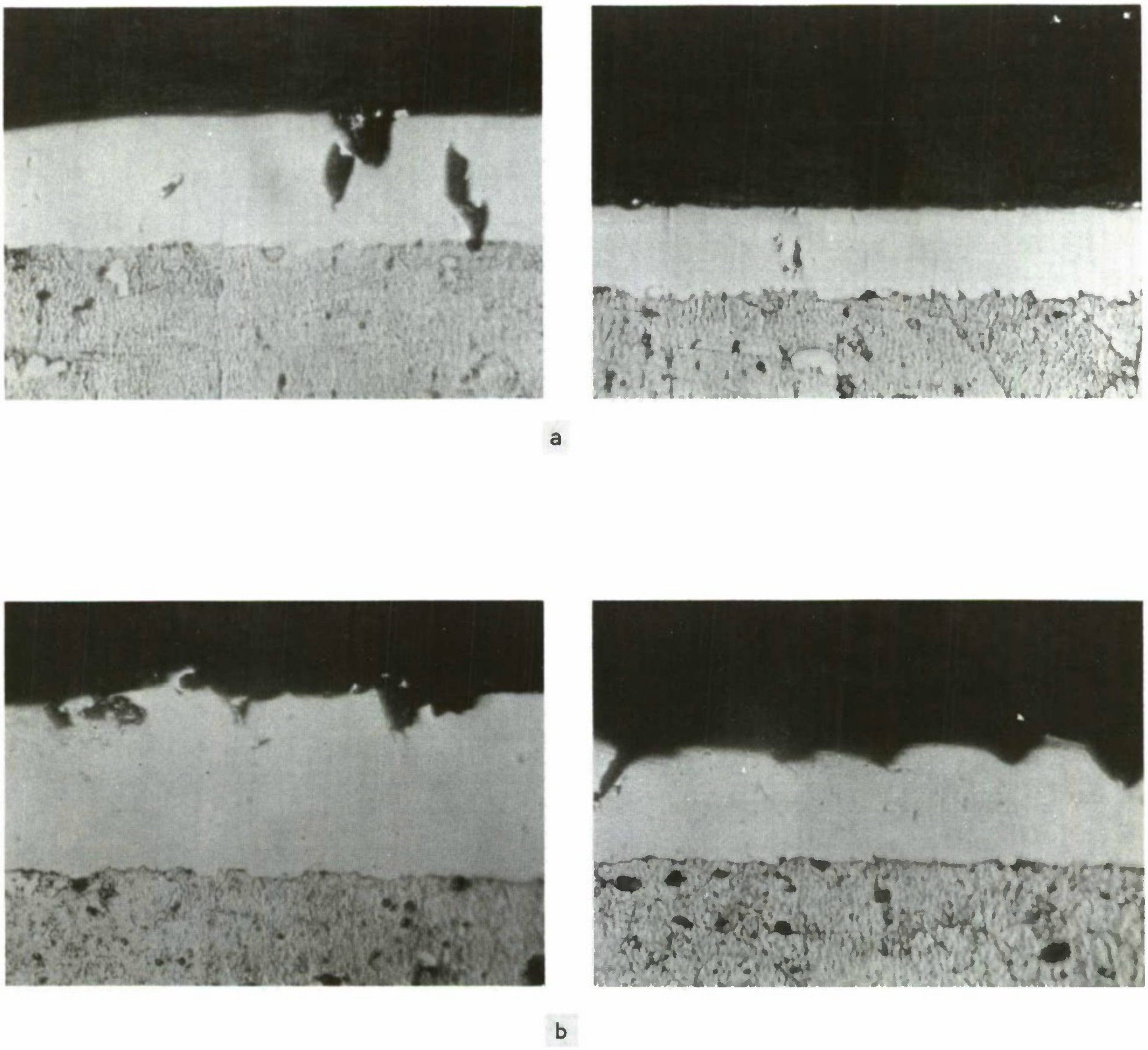


FIGURE 11 (#308-9) Addition of 50 g/l boric acid with (a) and without (b) PR, 120-6 second, effective cycle 40-1, 55°/50 asd in 100 g/l CrO_3 bath. Edge (left) and edge + 1.5cm (right).

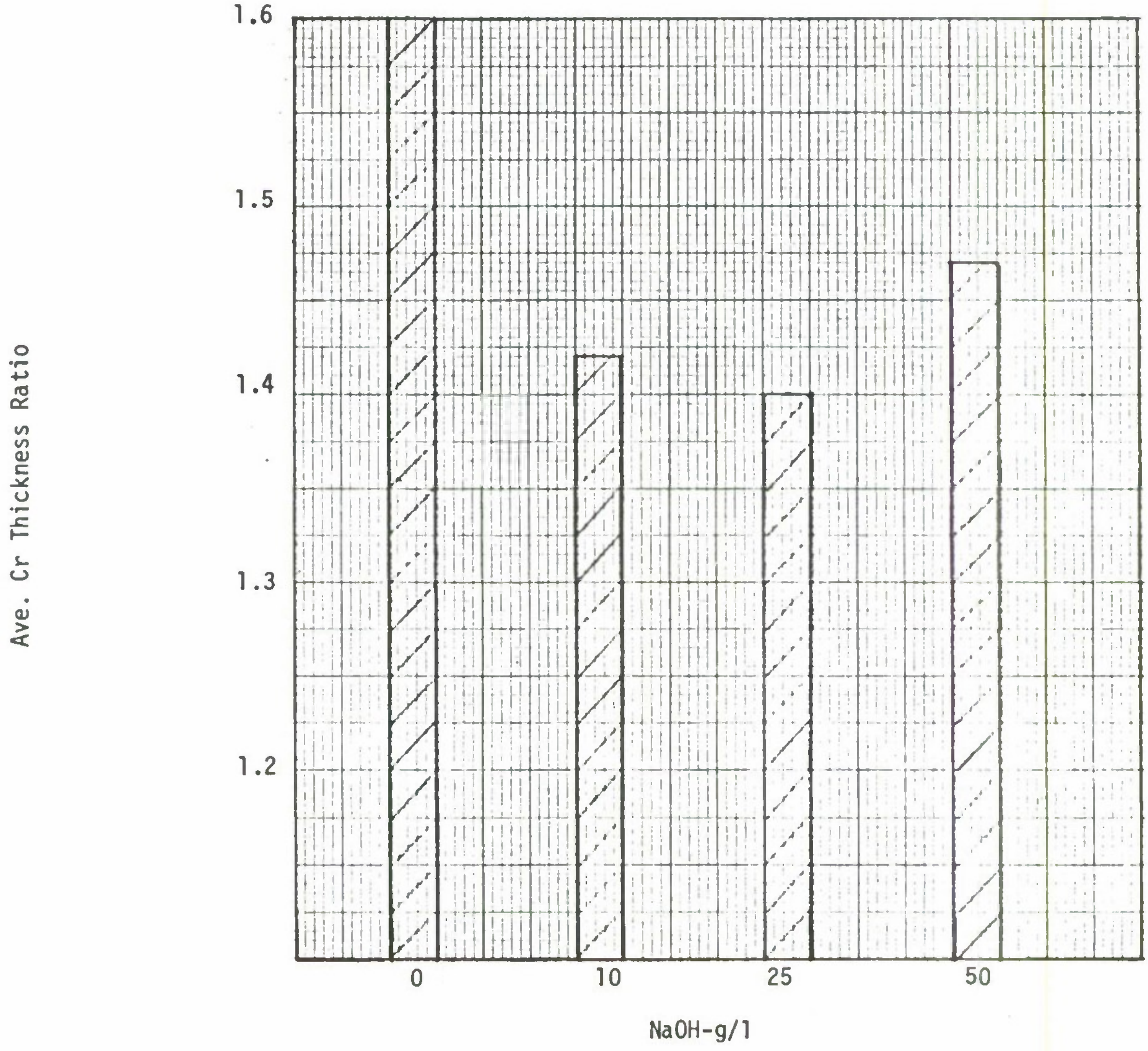


FIGURE 12 Sodium hydroxide added to 250 g/l CrO_3 bath for PR plating

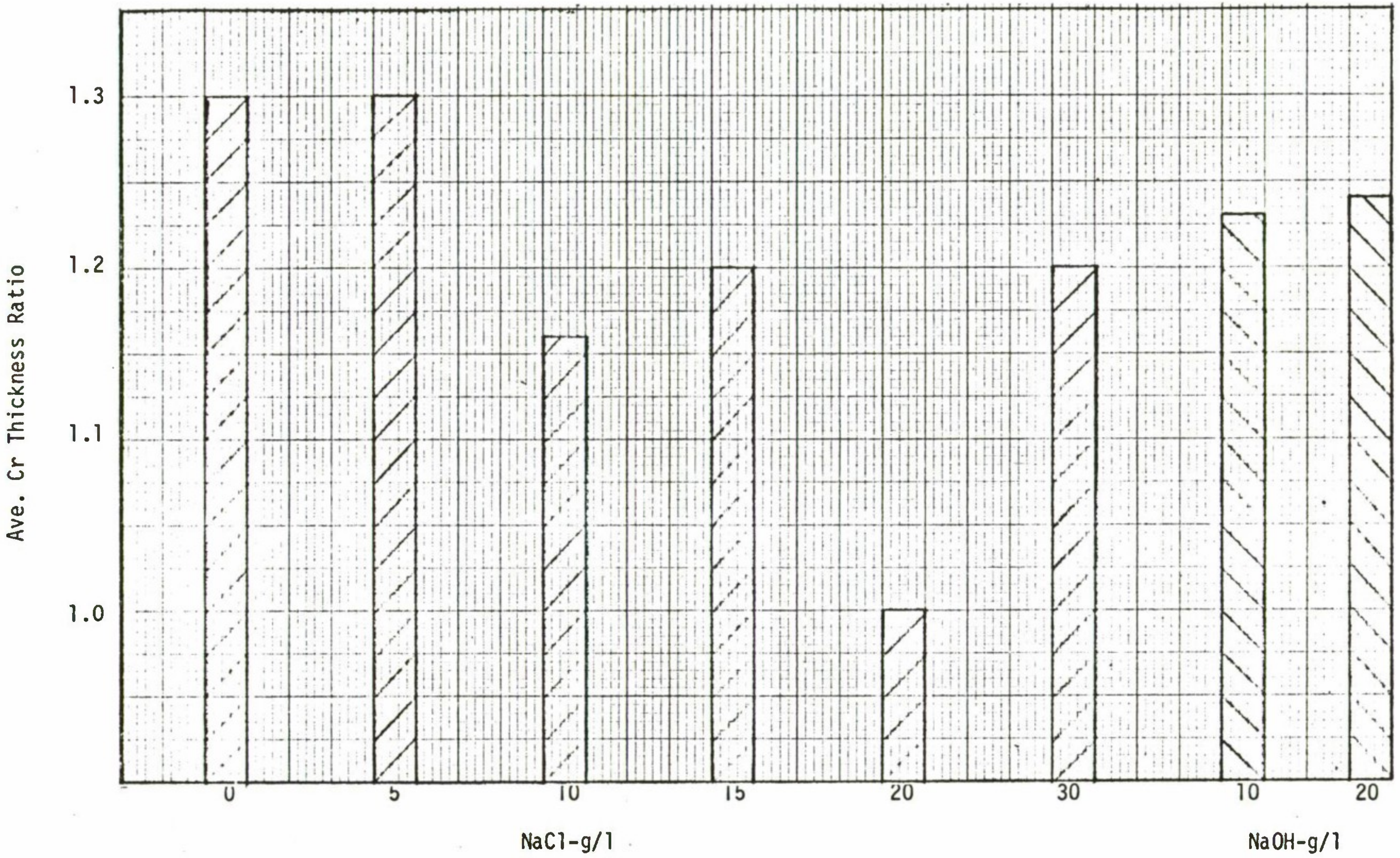


FIGURE 13 Sodium hydroxide and sodium chloride additions to separate 100 g/l CrO_3 baths for PR plating

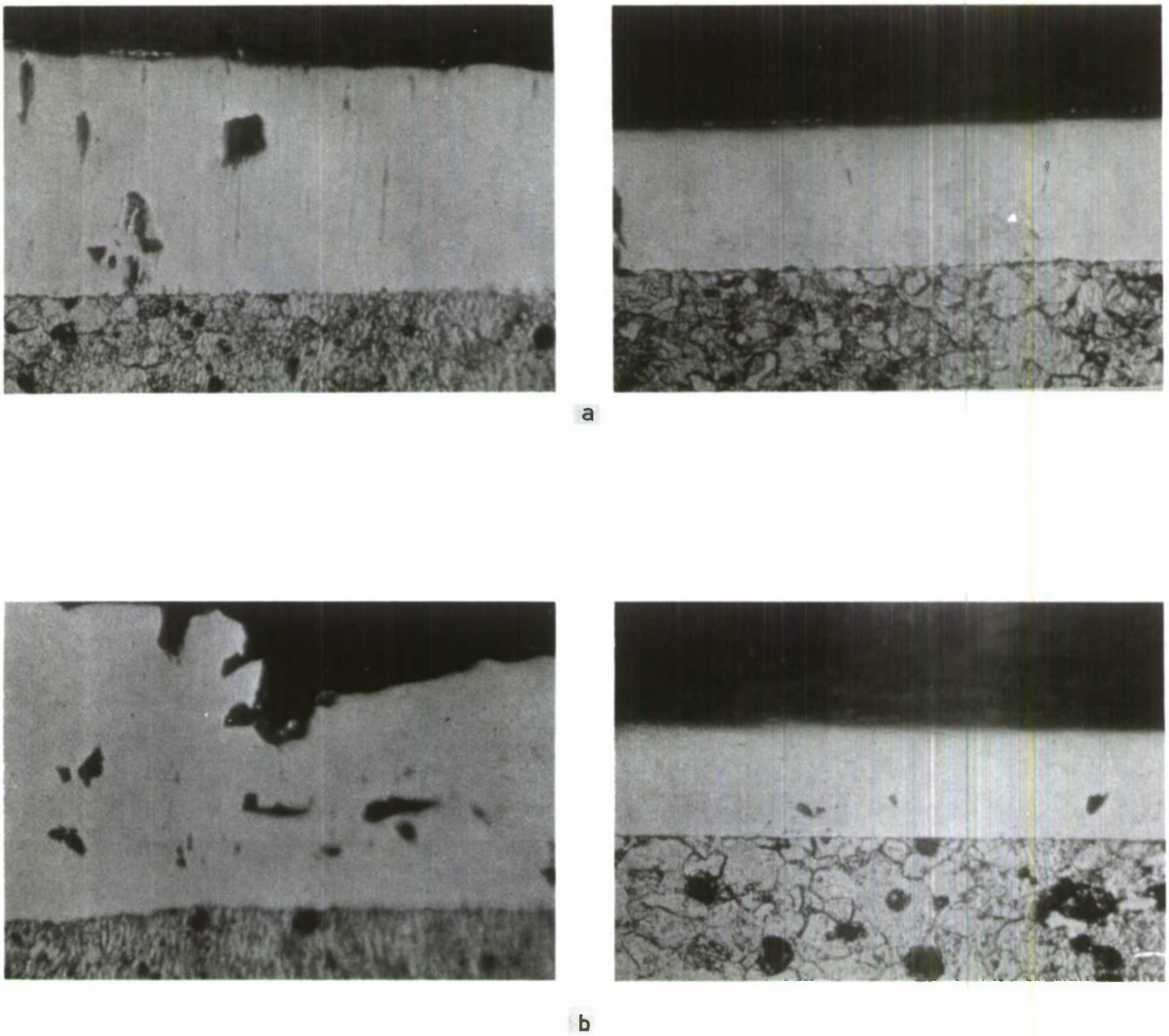
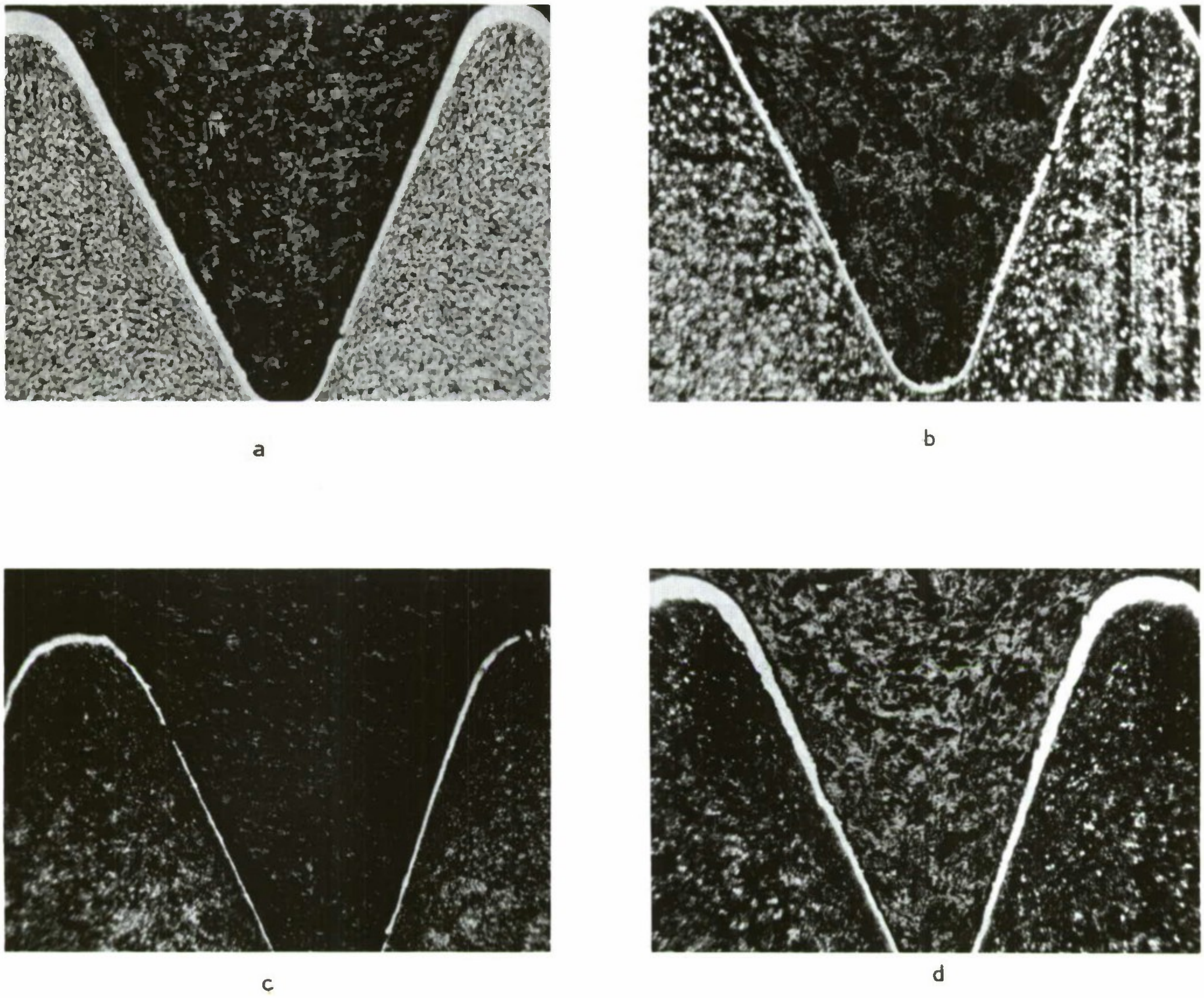


FIGURE 14 (#326-42) With (a) and without (b) addition of 25 g/l NaOH to 250 g/l CrO_3 bath 55°/50 asd. Edge (left) and edge + 1.5cm (right).



a

b

c

d

FIGURE 15 Cross-sections of Cr plated screw threads: a) 65°/30 asd, no PR; b) 120-3 sec, effective cycle 20-1, 65/30; c) same + NaOH additive; d) 120-5 sec, 20-1 effective cycle, 65/50. All in 250 g/l CrO_3. Approx. 50X.

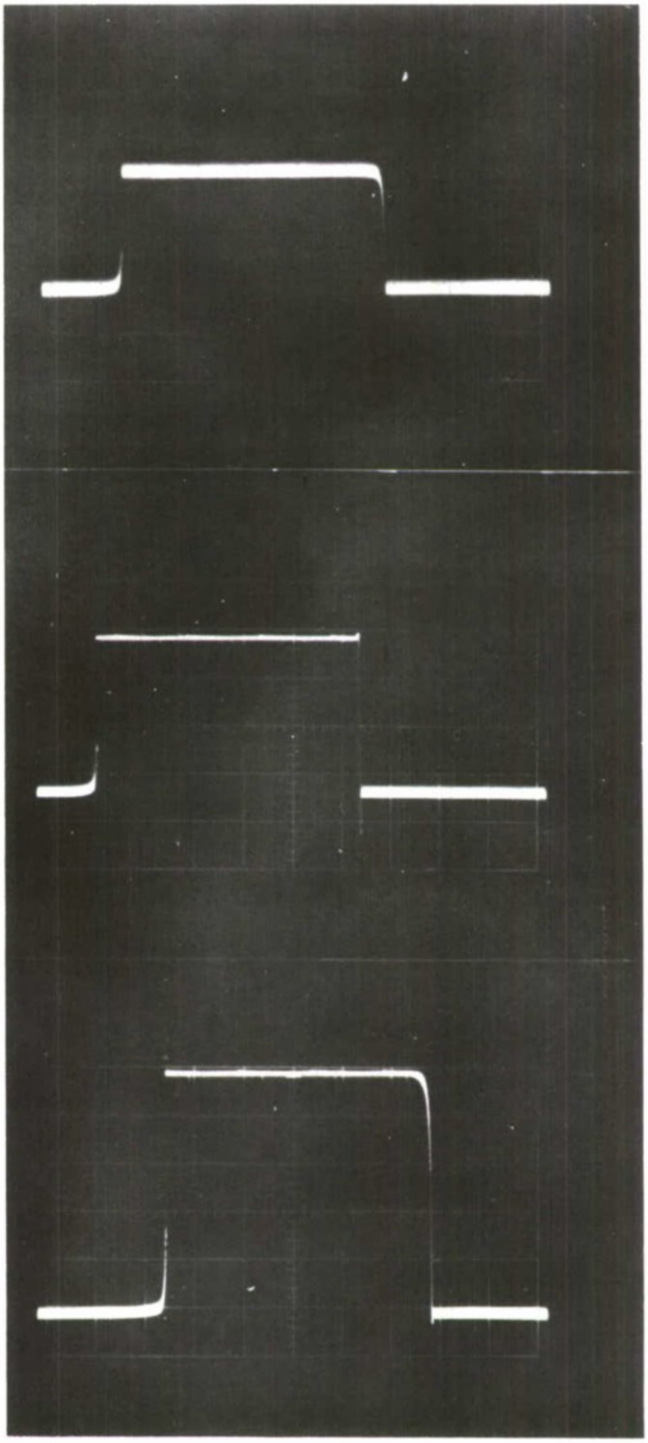


FIGURE 16 Pulse wave forms at (top) 50% base, 45 asd; (center) 25% base, 45 asd; (bottom) 25% base, 30 asd. 10 msec on, 10 off. Center pulse current controlled, others voltage controlled

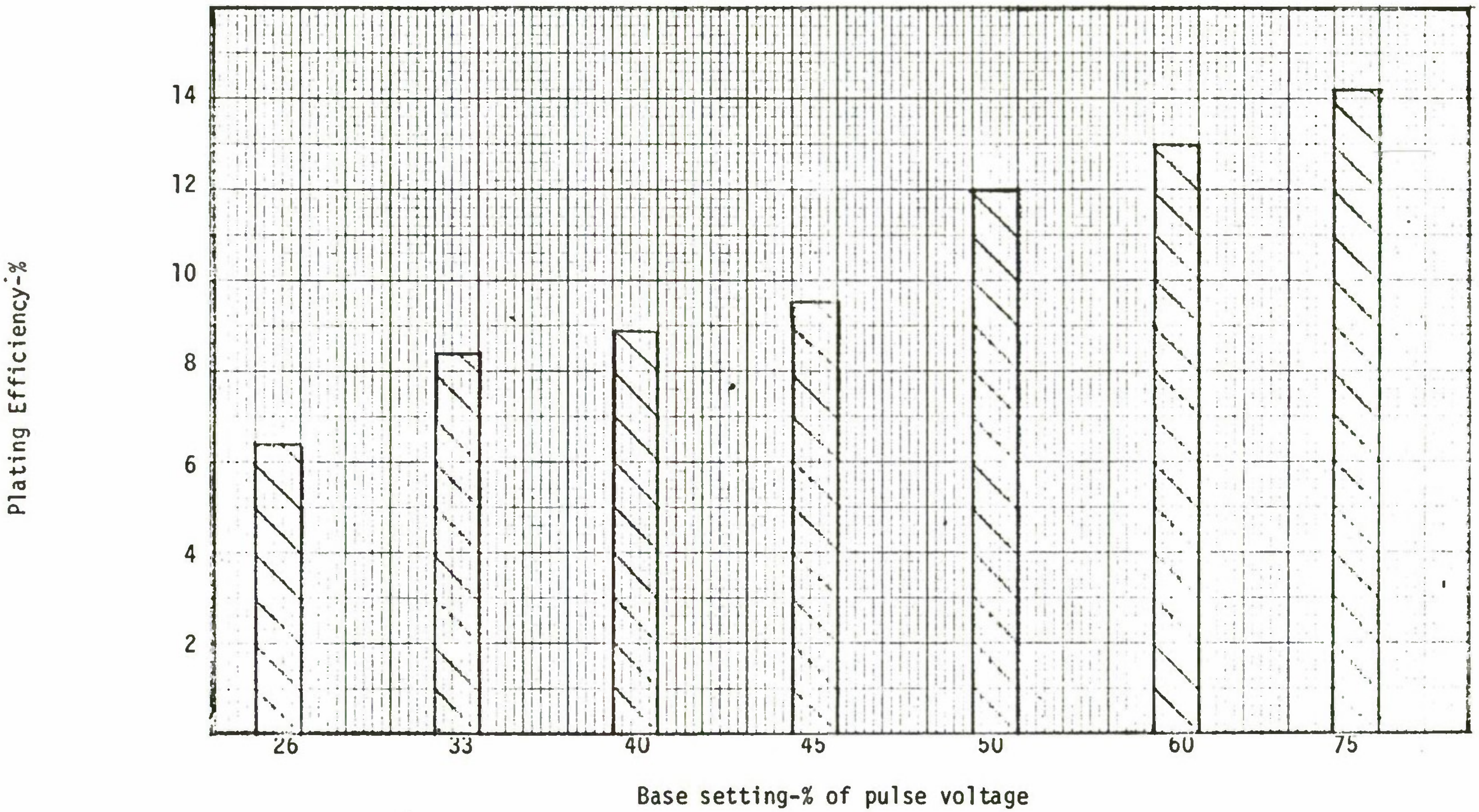


FIGURE 17 Between-pulse base current value as related to efficiency of average current applied to work

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APPENDIX A

Tabular Data

Cr PLATED THREADS

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle	Current Density For.-Rev. (asd)	Cr Thickness Ratio Top and bottom 1/4"-20" Threads
Dilute (100 g/1 CrO_3) Bath (50 and 55°C)				
1	120-3	40-1	50-50	4
2	120-6	20-1	"	4
3	No PR	--	50	3.5
4	120-9	15-1	50-50	4
Dilute Bath (65°C)				
16	120-3	20-1	30-60	3
17	No PR	--	30	3.5
18	120-16	15-1	50-25	4
Dilute Bath + 10 g/1 NaOH (50-55°C)				
5	120-6	20-1	50-50	7
6	"	40-1	50-25	8
7	120-3	20-1	50-100	2.3
8	"	"	30-60	2
9	"	"	"	4
10	No PR	--	30	2
11	120-6	40-1	30-15	2
12	"	"	20-10	1.5
13	120-9	25-1	30-15	2.5
14	"	"	50-25	4
Dilute Bath + NaOH (65°C)				
15	120-12	20-1	50-25	2.5
19	120-16	15-1	"	6
20	No PR	--	30	2.5
Std. Bath (250 g/1 CrO_3) 65°C				
21	No PR	--	30	2.7
22	120-3	80-1	30-15	3
23	"	20-1	30-60	1.3
24	120-6	20-1	50-50	4
25	60-3	"	"	3
26	120-6	"	30-30	2
27	30-1-1/2	"	50-50	4
28	120-5	"	50-65	2
29	40-2	"	50-50	4
Std. Bath + NaOH-65°C				
30	120-3	20-1	30-60	1.3
31	No PR	--	50	5
32	120-5	20-1	50-60	3.5
33	No PR	--	30	3
34	120-12	20-1	50-25	5
35	120-6	40-1	"	6

Test No.	Pulse On-Off (millisec)	Pulse Control (volts, amp)	Base Level (% of Pulse Voltage)	Temp - Current Density (asd)	Cr Surface	Cr Thickness Ratio Edge to Ctr. (mils)
533	90-10	V	5	65/30	Coarse matte	1.5
534	80-20	V	0	"	Matte	2.5
535	"	V	20	"	Satin	1.8
536	"	V	30	"	Brt satin	2.3
537	"	V	35	"	"	3.0
538	50-50	V	"	"	Semi-brt	2.8
539	"	V	50	"	"	3.0
540	"	V	25	"	Matte	2.2
541	"	V	"	65/45	Brt	2.0
542	"	V	"	65/60	Brt satin	1.3
543	10-10	V	50	65/45	Matte	1.5
544	"	V	"	"	Near-brt	2.1
545	"	V	25	"	Matte	1.5
546	"	A	"	"	Brt satin	2.0
547	None	-	100	"	Near-brt	3.1
548	"	-	"	65/30	"	2.6

Test No.	PR Cycle (sec.) for.-rev.	Effective Cycle** (sec)	Forward Current Density (asd)	Cr Surface	Cr Thickness (mil) Near Edge	Center	Far Edge	Ratio (Edge to Edge)
13	12-1/2	8-1	20	Dk.-partly bare	0.10	0.03	0	-
14	"	"	"	Matte	0.45	0.14	0.009	50
15	none	-	"	Dull	0.10	0.02	0.015	-
19	"	-	"	Satin	1.45	0.59	0.50	2.9
20	"	-	"	Semi-brite	1.16	0.52	0.52	2.2
21	12-1/2	8-1	"	Dk.-matte	0.14	0.06	0.04	3.5
22	12-1/4	24-1	"	Matte	0.27	0.10	0.06	4.5
23	"	27-1	"	"	0.39	0.10	0.06	6.5
24	"	"	"	"	0.32	0.09	0.05	6.4
25	"	24-1	"	Dk.-matte	1.03	0.17	0.09	11.5
26	none	-	"	Semi-brite	1.81	0.37	0.30	6
27	12-1/2	8-1	"	-	0	0	0	-
28*	18-1/2	12-1	"	Satin	1.89	0.22	0.17	11.1
29	"	"	"	Matte	0.24	0.14	0.06	4
30	18-1/4	27-1	"	Matte	0.90	0.45	0.36	2.5
31	"	24-1	"	"	1.63	0.56	0.33	4.9
32	18-1/8	96-1	"	"	1.66	0.83	0.87	1.9
33	none	-	"	Brite	1.46	0.49	0.47	3.1
34	18-1/8	96-1	"	Matte	1.32	0.59	0.53	2.5
35	24-1/8	128-1	"	"	1.36	0.67	0.61	2.2
36	12-1/8	70-1	30	"	1.49	0.79	0.28	5.3
37	"	"	"	"	1.28	0.39	0.23	5.6
38	30-1	11-1	20	"	1.50	0.13	0.01	150
39***	"	10-1	"	Dk.-matte	1.40	0.18	0.11	12.7
40	60-1	20-1	"	Satin	1.25	0.78	0.72	1.7
41	120-1	42-1	"	"	1.64	0.85	0.61	2.7
42	90-1	32-1	"	"	1.64	0.78	0.77	2.1
43	180-1	60-1	"	Semi-brite	1.40	0.72	0.73	1.9
44	none	-	"	Brite	1.77	0.72	0.61	2.9
45	22.5-1/8	90-1	"	"	1.93	0.70	0.66	2.9
46	180-2	35-1	"	Satin	1.77	0.68	0.65	2.7

*PR not working last 1/2 hr of plating time.

**Based on current differential between forward and reverse parts of cycle as shown on rectifier meter.

***New bath.

Test No.	PR Cycle For.-Rev. (sec)	Effective** Cycle	Forward Current Density (asd)	Cr Surface	Cr Thickness (mil)			Thickness Ratio
					Edge	Center	Edge	
Cycle Variation Tests								
47	180-3	22-1	20	Satin	1.50	0.51	0.56	2.7
48	180-4	17-1	"	"	1.50	0.59	0.54	2.8
49	180-5	13-1	"	Matte	1.30	0.48	0.48	2.7
50	180-5	"	"	Satin	1.85	0.57	0.58	3.2
51	180-6	11-1	"	"	1.41	0.40	0.49	2.9
52	180-8	8-1	"	Matte*	1.43	0.29	0.23	6.2
53	180-10	6.5-1	"	"*	1.20	0.27	0.24	5.0
54	90-5	"	"	"*	0.73	0.33	0.28	2.6
55	90-4	9-1	"	"*	1.98	0.50	0.38	5.2
56	45-2	8-1	"	"	1.50	0.38	0.40	3.8
57	22.5-1	9-1	"	"*	0.41	-	-	-
58	45-3	5.5-1	"	"*	1.14	-	-	-
59	60-3	8-1	"	"*	1.43	0.36	0.31	4.6
60	60-2	11-1	"	"	1.19	0.44	0.43	2.8
61	45-2	9-1	"	"	1.12	0.48	0.52	2.2
Bath CrO_3 Concentration Tests (300 g/1)								
62	45-3	5.5-1	20	Matte*	0.95	-	0.26	3.6
63	45-3	6-1	"	"	0.12	0.06	0.06	2.0
64	45-4	4-1	"	"*	0.37	-	0.13	2.8
65	45-4	4-1	"	"*	0.73	-	-	2.8
66	22.5-1.5	5.5-1	"	-*	-	-	-	-
67	22.5-1	8-1	"	Matte*	0.59	-	-	-
68	45-3	5.5-1	"	"*	0.94	-	-	-
69	45-2	8-1	"	"	1.39	0.32	0.25	5.5
400 g/1 CrO_3								
70	45.2	8:1	20	Matte	0.64	0.23	0.27	2.4
71	22.5-1	"	"	"	1.20	0.24	0.37	3.3
72	45-3	5.5-1	"	"*	0.03	-	-	-
73	45-3	"	"	-*	-	-	-	-
74	45-3	"	"	-*	-	-	-	-
75	60-3	7-1	"	Matte*	1.06	-	-	-
76	60-3	"	"	"*	0.82	-	-	-
77	90-3	11-1	"	"*	0.45	0.18	0.11	4.1
78	45-2	8-1	"	"*	1.05	-	-	-
79***	45-2	9-1	"	"*	1.09	0.22	0.17	6.4
80	60-3	7-1	"	"*	0.53	-	-	-
500 g/1 CrO_3								
81	60-3	7-1	20	-*	-	-	-	-
82	45-2	9-1	"	-*	-	-	-	-
83	60-2	11-1	"	Matte*	0.60	-	-	-
84	90-2	15-1	"	"	0.56	0.19	0.17	3.3
85	45-1	16-1	"	"	0.95	0.23	0.17	5.5
86	45-1.5	11.5-1	"	"*	0.60	-	-	-
87	45-1.5	12-1	"	"	0.96	0.21	0.13	7.4
88	45-1.5	10,5-1	30	"*	0.97	0.36	0.11	8.8
89	45-2	11-1	"	"*	1.25	0.30	-	-
90	22.5-1	11-1	"	"*	1.13	0.06	0.07	16
91	22.5-1	12-1	"	"*	1.51	0.36	-	-
92	22.5-1	12-1	"	"*	0.51	0.34	0.08	6.4
Parallel Cathode and Anode (500 g/1 CrO_3)								
93	45-2	11-1	30	Matte*	0.30	0.36	0.27	0.8
94	45-2	11-1	"	"*	0.47	0.44	0.25	1.1
95	-	-	"	Semi-Brt	1.12	0.58	0.92	1.9
96	45-1	22-1	"	Matte	1.22	0.57	0.92	2.1
97	60-1	30-1	"	"	0.91	0.61	0.96	1.5
98	90-1	45-1	"	Lt. Matte	1.01	0.67	1.35	2.0
99	90-2	22-1	"	"	0.86	0.56	0.60	1.5
100	180-4	23-1	"	Satin	0.84	0.50	0.67	1.7
101	180-6	15-1	"	"	0.70	0.55	0.69	1.3
102	180-8	11-1	"	"	0.56	0.41	0.32	1.4

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* Unplated area on specimen

** Based on current differential between forward and reverse parts of cycle as shown on rectifier meter at the same voltage setting

*** Bath filtered to remove fine sludge, mostly lead chromate.

Test No.	PR Cycle For.-Rev. (sec)	Effective** Cycle	Forward Current Density (asd)	Cr Surface	Cr Thickness (mil)			
					Edge-Ctr.(K)	Ratio	Edge-Ctr.(M)	Ratio
Specimen with back coated (C) and uncoated.								
95(C)	none	-	30	Semi-brt.	1.12-0.58	1.8	3.8-0.8	4.7
103	"	-	"	Lt. matte	1.40-0.92	1.5	7.5-2.2	3.4
116	22.5-1	10-1	"	Matte*	0.43-0.53	0.8	0.8-0.7	1.1
115	60-1/2	50-1	"	Satin	0.64-0.60	1.1	1.2-0.6	2.0
97(C)	60-1	30-1	"	Lt. matte	0.91-0.61	1.5	3.0-1.0	3.0
114	"	"	"	Satin	0.67-0.65	1.0	3.0-1.4	2.1
113	60-2	15-1	"	"	0.54-0.46	1.2	1.5-0.5	3.0
98(C)	90-1	45-1	"	Lt. matte	1.01-0.67	1.5	3.7-1.1	3.4
111	"	43-1	"	Satin	0.65-0.68	0.9	1.4-0.7	2.0
99(C)	90-2	22-1	"	Lt. matte	0.86-0.56	1.5	3.5-1.1	3.2
112	"	20-1	"	Satin	0.50-0.46	1.1	1.5-0.6	2.5
100(C)	180-4	23-1	"	"	0.84-0.50	1.7	3.0-1.1	2.7
106	"	22-1	"	"	0.43-0.34	1.3	2.2-0.8	2.7
101(C)	180-6	15-1	"	"	0.70-0.55	1.3	3.0-1.1	2.7
105	"	"	"	"	0.37-0.39	0.9	1.2-0.6	2.0
102(C)	180-8	11-1	"	"	0.56-0.41	1.4	2.0-0.7	2.9
104	"	"	"	Lt. matte*	0.33-0.35	0.9	1.0-0.8	1.3
133(C)	22.1-1	"	40	Matte	0.63-0.64	1.0	5.3-0.7	7.6
117	"	"	"	" *	0.73-0.81	0.9	0.8-0.7	1.1
129(C)	45-1	22-1	"	Satin	0.94-0.69	1.4	4.6-1.1	4.2
118	"	"	"	Lt. matte	1.21-0.88	1.4	6.4-1.2	5.3
119	45-2	11-1	"	" *	0.78-0.80	1.0	1.7-0.8	2.1
128(C)	60-1	30-1	"	Satin	1.12-0.95	1.2	4.0-1.1	3.6
122	"	"	"	" *	0.35-0.50	0.7	0.3-0.5	0.6
121	60-2	15-1	"	"	0.56-0.58	1.0	1.0-0.8	1.2
120	60-3	12-1	"	Lt. matte*	0.30-0.36	0.8	0.3-0.4	0.8
130(C)	90-2	23-1	"	Satin	0.71-0.40	1.8	3.2-0.5	6.4
124	90-3	15-1	"	"	0.63-0.72	0.9	2.0-1.0	2.0
123	90-4.5	10-1	"	"	0.82-0.78	1.1	2.6-1.4	1.9
127	120-2	30-1	"	"	1.40-0.73	1.9	1.3-1.2	1.1
126	120-4	15-1	"	"	0.63-0.65	0.9	1.1-1.1	1.0
125	120-6	10-1	"	"	0.53-0.47	1.3	0.6-0.8	0.8
Current Density Test (500 g/1 CrO_3)								
140	22.5-1	8-1	20	-	-	- (no Cr)	-	-
141	45-1	15-1	"	Satin	0.52-0.35	1.5	1.6-0.5	3.2
142	60-1	20-1	"	"	0.62-0.34	1.8	2.4-0.7	1.4
143	60-2	10-1	"	"	0.37-0.25	1.5	1.7-0.7	2.4
139	"	"	"	"	0.38-0.24	1.6	1.0-0.6	1.7
96	45-1	22-1	30	Matte	1.22-0.57	2.1	3.4-0.6	5.7
94	45-2	11-1	"	"	0.47-0.44	1.1	1.1-0.3	3.7
97	60-1	30-1	"	Lt. matte	0.91-0.61	1.5	3.0-1.0	3.0
98	90-1	45-1	"	"	1.01-0.67	1.5	3.7-1.1	3.4
99	90-2	22-1	"	Lt. matte	0.86-0.56	1.5	3.5-1.1	3.2
100	180-4	"	"	Satin	0.84-0.50	1.7	3.0-1.1	2.7
101	180-6	15-1	"	"	0.70-0.55	1.3	3.0-1.1	2.7
102	180-8	11-1	"	"	0.56-0.41	1.4	2.0-0.7	2.9
133	22.5-1	11-1	40	Matte	0.63-0.64	1.0	5.3-0.7	7.6
129	45-1	22-1	"	Satin	0.94-0.69	1.4	4.6-1.1	4.2
128	60-1	30-1	"	"	1.12-0.95	1.2	4.0-1.1	3.6
130	90-2	22-1	"	"	0.71-0.40	1.8	3.2-0.5	6.4
131	180-6	15-1	"	"	1.06-0.65	1.6	5.2-1.7	3.1
132	180-8	11-1	"	"	0.67-0.54	1.2	4.0-0.7	5.7
134	22.5-1	11-1	50	Matte	0.61-0.43	1.4	2.4-2.2	1.1
135	45-1	20-1	"	Satin	0.99-0.87	1.1	3.7-0.8	4.6
136	60-1	25-1	"	"	1.55-0.90	1.7	5.9-1.2	4.9
138	60-2	13-1	"	"	1.42-0.92	1.5	5.6-0.8	7.0
137	90-2	20-1	"	"	1.17-0.94	1.3	4.3-1.0	4.3

*Unplated area on specimen

**Based on current differential between forward and reverse parts of cycle as shown on rectifier meter at a given voltage setting.

NOTE: All runs were made in a 500g/1 CrO_3 bath with 100-1 sulfate.

K = Kocour thickness measurement.
M = Thickness measurement by micrometer.

Test No.	PR Cycle For.-Rev. (sec.)	Effective Cycle**	For.-Rev. Current Density (asd)	Cr Surface	Cr Thickness	
					Edge-Ctr. (K) Ratio	Edge-Ctr. (M) Ratio
			Current Density Test Cont.			
138	60-2	13-1	50-116	Satin	1.3	5.6
139	60-2	10-1	20-60	Satin	1.1	1.5
140	22.5-1	7-1	20-60	No Cr	---	---
141	45-1	16-1	20-55	Satin	2.4	6.6
142	60-1	21-1	20-59	Satin	1.5	3.0
143	60-2	10-1	20-60	Satin	1.4	2.8
144	none	----	50-	Brt	1.8	4.3
145	none	----	40-	Semi-Brt	1.8	2.5
			Controlled Reverse Current			
146	180-16	11-1	30-30	Satin	1.5	2.2
147	120-12	10-1	30-30	Satin	1.3	5.8
148	90-9	10-1	30-30	Lt matte*	---	---
149	90-6	15-1	30-30	Lt matte*	1.1	5.3
150	90-9	10-1	30-30	Satin*	---	4.5
151	180-18	10-1	30-30	Satin*	1.8	6.4
152	60-4	15-1	30-30	Lt matte	1.3	3.1
153	60-6	10-1	30-30	Lt matte*	1.4	3.1
154	45-4	11-1	30-30	Lt matte*	1.7	3.7
155	22.5-2	11-1	30-30	Matte*	1.5	2.3
156	11-1	11-1	30-30	Satin*	2.4	3.3
157	10-1	10-1	30-30	Matte*	3.1	4.3
158	180-18	10-1	30-30	Satin	1.4	2.5
			Low Reverse Current			
159	180-36	10-1	30-15	Lt matte	0.7	1.0
179	180-30	12-1	30-15	Satin	1.2	3.4
180	180-24	15-1	30-15	Satin	1.3	8.4
181	180-24	15-1	30-15	Satin	1.4	4.7
160	90-18	10-1	30-15	Satin	1.5	2.2
161	60-12	10-1	30-15	Satin	1.0	4.8
162	45-9	10-1	30-15	Lt matte*	1.0	13.0
163	45-9	10-1	30-15	Matte*	1.0	4.5
164	60-12	10-1	30-15	Matte*	1.4	10.0
165	none	----	30-	Brt	2.8	7.3
166	none	----	30-	Brt	1.8	11.3
			Cycle-Length Test			
167	60-9	13-1	30-15	Satin	1.5	3.7
168	60-6	20-1	30-15	Satin	1.3	6.3
169	60-3	40-1	30-15	Satin	1.4	5.9
170	90-13.5	13-1	30-15	Satin	1.1	4.9
171	90-9	20-1	30-15	Satin	1.3	4.8
172	90-4.5	40-1	30-15	Satin	1.2	4.1
173	120-12	20-1	30-15	Satin	1.2	6.6
174	120-6	40-1	30-15	Satin	1.3	6.5
175	120-3	80-1	30-15	Satin	1.3	7.1
176	180-18	20-1	30-15	Satin	1.2	5.4
177	180-9	40-1	30-15	Satin	1.3	3.6
178	180-4.5	80-1	30-15	Satin	1.3	5.1
182	150-30	10-1	30-15	Satin	1.7	5.3
183	150-22.5	13-1	30-15	Satin	2.8	7.3
184	150-15	20-1	30-15	Satin	1.3	3.4
			High Current Reverse			
185	90-4.5	10-1	30-60	Matte	2.0	3.2
186	90-3	13-1	30-68	Matte	1.6	2.2
187	180-9	9-1	30-68	Satin*	1.1	5.1
188	180-4.5	10-1	30-120	Lt Matte*	1.6	10.0
189	180-[illegible]	12-1	30-150	Satin	1.3	3.9
190	60-0.3	40-1	30-150	Lt Matte	1.4	5.6
191	60-1	11-1	30-165	Matte*	1.2	1.9

* Unplated area on specimen

** Based on current difference between forward and reverse parts of cycle.

Note: All runs made in 500 g/l CrO$_3$ bath with 100-1 sulfate.
K = Kocour thickness measurement
M = Micrometer measurement at extreme edges and central area of specimen.

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Ratio		
					Edge-Ctr.	(K) (mil)	Edge-Ctr. (M)
High Current Reverse (cont.)							
194	60-2	6-1	30-165	Satin*	0.008-0	—	--
195	120-2	12-1	30-165	Lt. Matte	0.58-0.46	1.3	3.0
196	120-1.5	16-1	30-165	Satin	0.66-0.45	1.5	7.7
228	120-2	15-1	20-85	—	No Cr		
229	120-1	30-1	20-85	--	Cr at extm. edges only		
230	120-1	40-1	30-90	Matte	0.71-0.44	1.6	5.7
231	120-2	15-1	30-120	Satin*	0.39-0.15	2.6	—
232	120-2	15-1	40-160	Satin*	1.17-0.41	2.6	3.9
233	120-1	30-1	40-160	Matte	1.30-0.48	2.7	4.9
234	120-1/2	60-1	40-160	Matte	2.02-0.70	2.9	4.7
235	120-1/2	120-1	50-100	Lt. Matte	2.25-0.73	3.1	4.0
236	120-1	60-1	50-100	Matte	1.86-0.70	2.7	4.3
237	120-2	30-1	50-100	Lt. Matte	2.21-0.71	3.1	3.9
238	120-2	30-1	50-150	Matte	1.68-0.62	2.7	4.4
239	120-3	13-1	50-150	Satin	0.66-0.35	1.9	6.4
Low Current Reverse (Cont.)							
197	45-4.5	20-1	30-15	Lt. Matte	0.81-0.49	1.7	9.2
198	45-2	45-1	30-15	Lt. Matte	1.55-0.76	2.0	8.2
199	45-2	45-1	30-15	Satin	1.01-0.66	1.5	12.0
200	45-1	90-1	30-15	Satin	0.93-0.59	1.6	9.3
201	22.5-2	22-1	30-15	Matte	0.64-0.45	1.4	4.0
202	22.5-1	45-1	30-15	Satin	1.32-0.78	1.7	4.5
203	22.5-5	90-1	30-15	Ok. Satin	1.18-0.90	1.3	2.4
204	10-1	20-1	30-15	Ok. Satin	0.86-0.35	1.5	5.7
205	10-.5	40-1	30-15	Ok. Satin	1.02-0.73	1.4	4.5
206	10-.25	80-1	30-15	Ok. Satin	1.06-0.65	1.6	12.3
207	10-.25	80-1	40-20	Ok. Satin	0.75-0.55	1.4	5.0
208	10-.5	40-1	40-20	Ok. Satin	1.58-0.84	1.9	5.0
209	10-1	20-1	40-20	Matte	0.91-0.67	1.4	6.3
210	22.5-2	20-1	40-20	Satin	0.63-0.33	1.9	4.3
211	10-2	10-1	40-20	--	(No Cr)	--	--
212	22-1	40-1	40-20	Satin	0.99-0.50	2.0	7.1
213	45-1	90-1	40-20	Satin	1.32-0.67	2.0	10.2
214	45-2	45-1	40-20	Satin	0.76-0.48	1.6	9.0
215	45-4.5	20-1	40-20	Satin	0.85-0.48	1.8	7.5
216	45-9	10-1	40-20	Satin*	0.54-0.27	2.0	17.2
217	90-18	10-1	40-20	Satin*	0.35-0.25	1.3	10.0
218	120-24	10-1	40-20	Matte*	0.26-0.09	2.9	7.0
219	180-30	10-1	40-20	Satin*	0.52-0.23	2.3	18.0
220	90-9	20-1	40-20	Satin*	1.22-0.73	1.7	6.8
221	120-24	10-1	20-10	Satin*	0.006	--	--
222	120-12	20-1	20-10	Satin*	0.49-0.34	1.4	1.6
223	120-6	40-1	20-10	Satin*	0.73-0.40	1.8	7.3
224	No PR	--	20-10	Lt. Satin	0.68-0.44	1.5	6.0
225+	120-3	80-1	20-10	Satin	0.52-0.31	1.7	2.0
226+	120-3	80-1	20-10	Satin	0.53-0.35	1.5	4.4
227	No PR	--	20-[illegible]	Satin	0.74-0.47	1.6	5.2
Edge Mounted							
240	120-3	13-1	50-150	Matte	0.66-0.32	2.1	8.3
241	120-4	10-1	50-150	Semi-brite	0.03-0.02	1.5	13.0
242	120-1	40-1	50-150	Matte	1.18-0.71	1.7	6.8
243	120-2	20-1	50-150	Satin	0.84-0.60	1.4	9.4
244	120-1	30-1	40-160	Satin	0.77-0.49	1.6	10.7
245	120-2	15-1	40-160	Satin	1.34-0.77	1.7	7.0

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Ratio Edge-Ctr. (K) (mil)		Edge-Ctr. (M)
			Plus 5 g/l Oxalic Acid				
246	120-1	40-1	30-90	Semi-brite	0.84-0.60	1.4	5.2
247	120-1/2	60-1	40-160	Satin	0.94-0.73	1.3	7.3
248	120-2	15-1	40-160	Satin	0.83-0.44	1.9	8.1
249	120-2	20-1	50-150	Matte	1.08-0.74	1.5	4.0
250	120-1	40-1	50-150	Matte	1.27-1.18	1.1	2.8
251	120-3	40-1	30-30	Satin	0.96-0.66	1.5	8.3
252	120-2	60-1	30-30	Satin	0.94-0.75	1.3	3.2
253	120-6	40-1	30-15	Satin	0.85-0.70	1.2	4.6
			Plus 10 g/l Oxalic Acid				
254	120-6	40-1	30-15	Satin	1.01-0.73	1.4	2.9
255	120-3	40-1	30-30	Satin	0.94-0.72	1.3	2.5
256	120-2	60-1	30-30	Satin	1.22-0.99	1.2	4.5
257	No PR	--	30	Brt	1.41-1.04	1.4	4.8

* Unplated area on specimen

** Based on current difference between forward and reverse parts of cycle

\+ New bath

Note: All runs made in 500 g/l CrO_3 bath with 100-1 sulfate.
K = Kocour thickness measurement.
M = Micrometer measurement at extreme edges and central area of specimen.

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Ratio Edge-Ctr (K) (mil)		(M)
		Plus 10 g/l oxalic acid (cont.)					
258+	No PR	--	50	Satin	2.91-1.91	1.6	2.8
259	"	--	40	Brt	1.92-1.20	1.6	9.0
260+	120-1	40-1	30-90	Semi-Brt	1.33-0.92	1.4	4.7
261	"	"	"	"	1.66-0.82	2.0	3.1
262	60-1	20-1	"	Lt. Matte	1.01-0.71	1.4	3.4
263+	"	"	"	Satin	0.80-0.69	1.2	3.7
264+	"	"	"	"	0.96-0.54	1.8	1.7
265	"	"	40-120	"	1.13-1.04	1.1	3.1
266+	"	"	"	"	1.04-0.77	1.5	2.0
267+	"	"	"	"	0.80-0.49	1.6	7.6
268	45-1	15-1	"	"	0.84-0.59	1.4	1.5
269+	"	"	"	"	0.66-0.50	1.3	1.6
270	120-2	20-1	"	"	0.85-0.67	1.3	2.8
271+	60-1.5	"	50-100	"	0.53-0.43	1.2	1.6
272	40-1	"	"	"	0.66-0.43	1.5	2.3
273+	20-1	10-1	"	No deposit			
274	30-1	15-1	"	Satin*	0.42-0.32	1.3	5.5
275+	30-4	15-1	50-25	Matte	0.58-0.50	1.1	7.1
276	40-4	20-1	"	"	0.82-0.58	1.4	4.1
277+	120-6	40-1	"	"	0.92-0.66	1.4	2.0
278	"	"	30-15	"	0.76-0.63	1.2	1.8
			Plus 20 g/l oxalic acid				
279+	120-2	20-1	30-90	Satin	0.86-0.65	1.3	1.6
280	60-1	"	"	"	0.60-0.50	1.2	2.0
281	45-1	15-1	"	"	0.69-0.47	1.5	2.5
282	60-1	20-1	"	"	0.55-0.49	1.1	1.4
283+	30-4	15-1	50-25	"	0.95-0.72	1.3	2.1
284	40-4	20-1	"	"	1.22-1.15	1.1	1.8
			Dilute bath - 100 g/l CrO_3				
285	30-4	15-1	50-25	No SO_4 addition-No Cr			
286	40-4	20-1	"	Lt. Matte	2.91-1.89	1.5	1.7
287	30-4	15-1	"	"	1.89-1.64	1.1	1.8
288	120-6	40-1	"	Semi-brt	2.27-2.20	1.0	2.2
			Dilute bath + 10 g/l oxalic acid				
289	120-6	40-1	50-25	Satin	2.14-2.33	0.9	1.7
290	30-4	15-1	"	Blk.	0.68-1.96	0.3	1.2

\+ Additions of oxalic acid

* Unplated area on specimen

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Edge-Ctr (mil)	Ratio (K)	(M)
Dilute Bath (100 g/l CrO_3) + 10 g/l Oxalic Acid							
291	40-4	20-1	50-25	Lt. Matte	1.38-1.20	1.1	1.4
292*+	45-1	15-1	30-90	Black	0.68-0.37	2.1	6.4
293	60-1	20-1	"	Matte	1.51-1.31	1.2	2.3
294*+	120-2	"	"	Lt. Matte	2.27-1.95	1.3	1.7
295	120-12	"	30-15	"	1.78-1.17	1.5	1.6
296	40-4	"	50-25	"	2.67-2.49	1.1	1.2
298	120-6	40-1	"	Satin	2.84-2.59	1.1	1.3
286	40-4	20-1	"	Lt. Matte	2.91-1.89	1.5	1.7
287	30-4	15-1	"	"	1.89-1.64	1.2	1.8
288	120-6	40-1	"	Semi Brt	2.27-2.20	1.0	2.2
Dilute Bath + 5 g/l Boric Acid							
299*	120-6	40-1	30-15	Satin	2.27-1.36	1.7	2.5
300	120-12	20-1	"	"	1.96-1.31	1.5	2.7
301*	120-6	40-1	50-25	Brt. Satin	2.29-1.72	1.3	1.6
302	40-4	20-1	"	Lt. Matte	2.00-1.66	1.2	2.2
10 g/l Boric Acid							
303*	120-6	40-1	50-25	Satin	1.89-1.47	1.3	1.6
304	40-4	20-1	"	Lt. Matte	1.87-1.51	1.2	1.8
305*	120-6	40-1	30-15	Satin	1.31-1.00	1.3	2.4
20 g/l Boric Acid							
306	120-6	40-1	30-15	Lt. Matte	1.48-1.17	1.3	--
307	--	--	30-	Brt	1.55-1.09	1.4	3.0
50 g/l Boric Acid							
308	--	--	50-	Brt	2.14-1.82	1.2	2.0
309*	120-6	40-1	50-25	Brt. Satin	1.42-1.19	1.2	1.9
310	180-8	40-1	"	"	1.61-1.26	1.3	1.8
311	40-4	20-1	"	Matte	1.59-1.17	1.4	1.5
100 g/l Boric Acid							
312*	120-6	20-1	50-25	Lt. Matte	1.68-1.17	1.4	1.5
313	120-2	"	30-90	"	1.16-0.94	1.2	1.8
314	60-1	"	"	Dk. Matte	1.56-0.50	3.1	4.4
250 g/l CrO_3, 50 g/l Boric Acid							
315	60-1	20-1	30-90	Matte	0.33-0.23	1.4	2.8
316	120-2	"	"	Lt. Matte	1.18-0.90	1.3	2.7
317	120-6	40-1	50-25	"	2.11-1.74	1.2	1.6
318	40-4	20-1	"	Matte	2.45-1.84	1.3	2.6
319	--	--	50-	Brt	1.59-1.03	1.5	2.2
320*	120-12	20-1	50-25	Satin	1.78-1.36	1.3	2.3
321	180-9	40-1	"	"	2.07-1.75	1.2	2.4
322	--	--	20-	Brt	1.54-0.98	1.6	4.1
323	60-2/3	20-1	50-100	Lt. Matte	1.69-1.27	1.3	3.2
324	120-1-1/3	"	"	Satin	1.55-1.07	1.4	2.4
325	120-2/3	40-1	"	"	2.59-1.97	1.3	2.7
250 g/l CrO_3 Only				No PR			
326	--	--	50-	Brt	2.62-1.89	1.4	3.5
327	--	--	30-	"	2.29-1.28	1.8	3.4

* Make-up addn. of CrO_3
\+ Make-up addn. of Oxalic Acid

Note: K=Kocour thickness measurement.
M=Micrometer measurement at extreme edges and central area of specimen.

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Edge-Ctr. (mil)	Ratio (K)	(M)
10 g/1 NaOH added to 250 g/1 CrO_3 Bath							
328	---	---	30	Brt	2.03-1.34	1.5	2.9
329	120-2	20-1	30-90	Satin	1.85-1.09	1.7	2.8
330	60-1	"	"	Lt. Matte	1.77-1.05	1.7	1.5
331	60-6	"	30-15	Satin	1.65-1.08	1.5	3.0
332	"	"	50-25	"	2.25-1.86	1.2	3.4
333	120-6	40-1	"	"	2.81-2.27	1.2	2.7
334	"	"	30-15	"	1.79-1.22	1.5	3.2
25 g/1 NaOH							
335	120-6	40-1	30-15	Satin	1.71-1.10	1.6	3.2
336	"	"	50-25	"	1.99-1.60	1.2	2.3
337	60-6	20-1	"	"	1.67-1.39	1.2	2.4
338	"	"	30-15	Lt. Matte	1.60-0.98	1.6	3.4
339	"	"	30-90	Matte	1.69-1.02	1.7	3.3
340	120-1	40-1	"	Satin	1.44-1.05	1.4	1.8
341	---	---	30	Brt	1.82-1.48	1.2	2.0
342	---	---	50	"	3.02-2.30	1.3	1.8
50 g/1 NaOH							
343	---	---	50	Brt$^+$	2.57-2.04	1.3	1.6
344	---	---	30	"$^+$	1.65-1.51	1.1	1.6
345	---	---	20	"$^+$	1.41-1.19	1.2	1.1
346	120-1	40-1	30-90	Matte$^+$	1.56-1.21	1.3	1.1
347*	60-1	20-1	"	"$^+$	2.30-1.51	1.5	1.4
348	60-6	"	30-15	"$^+$	1.59-0.85	1.9	1.7
349	120-6	40-1	"	"$^+$	1.85-1.16	1.6	1.3
350	"	"	50-25	Satin$^+$	2.79-1.86	1.5	2.1
351	60-6	20-1	"	Matte$^+$	3.03-2.13	1.4	1.2
352	40-4	"	"	"$^+$	2.39-1.29	1.9	1.7
100 g/1 CrO_3 Bath-(no additive)							
353	---	---	30	Brt	2.12-1.65	1.3	1.9
354	---	---	20	"	1.50-1.02	1.5	2.9
355	---	---	50	"	3.32-2.77	1.2	1.5
356	120-6	40-1	50-25	Satin	3.19-2.08	1.5	2.4
357*	60-6	20-1	"	"	2.46-1.92	1.3	1.4
358	40-4	"	"	"	2.71-1.49	1.8	1.8
359	120-1.5	40-1	30-60	Brt Satin	1.90-1.12	1.7	1.7
360	60-1.5	20-1	"	Dk. Matte	1.21-0.69	1.8	2.6
361*	60-1	"	30-90	Matte	1.97-1.53	1.3	2.1

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Edge-Ctr. (mil)	Ratio (K)	(M)
362	60-6	20-1	30-15	Blk	1.74-0.69	2.5	3.0
363*	120-6	40-1	"	Satin	1.25-0.94	1.3	2.7
364	40-4	20-1	"	Blk	0.55-0.21	2.6	5.8
365	120-1	40-1	30-90	Satin	2.10-1.65	1.3	1.7
Forward + Reverse Current Equal (100 g/1 CrO_3)							
366*	120-3	40-1	30-30	Lt. Matte	2.29-1.82	1.3	1.8
367	60-3	20-1	"	Dk. Matte	1.71-1.24	1.4	2.1
368	40-2	"	"	Blk	0.23-0.15	1.5	4.8
369	"	"	50-50	Matte	2.63-2.03	1.3	1.8
370	60-3	"	"	Satin	2.67-2.08	1.3	1.8
371*	120-3	40-1	"	"	2.52-2.02	1.3	1.5
372	180-9	20-1	"	"	2.47-2.20	1.1	1.1
Temperature Test (100 g/1 CrO_3)							
373*	120-3	40-1	(45°) 30-30	Lt. Matte	1.74-1.04	1.7	2.8
374	"	"	(40°) "	Matte	2.48-1.89	1.3	2.0
375	"	"	(35°) "	Dk. Matte	2.51-2.09	1.2	1.7
376*	"	"	(60°) "	Brt Satin	1.62-1.68	1.4	2.8
377*	"	"	(55°) "	Satin	2.12-1.60	1.3	2.0
378	"	"	(65°) "	Nr. Brt	1.92-1.13	1.7	2.5
379*	"	"	(70°) "	Semi-Brt	1.87-1.06	1.8	2.9
380	"	"	(80°) "	Satin	1.76-1.02	1.7	4.2
High Temperature (65°) PR Test (100 g/1 CrO_3)							
381	180-4-1/2	40-1	30-30	Nr. Brt	1.93-1.15	1.7	2.1
382	60-1-1/2	"	"	Brt Satin	2.29-1.36	1.7	2.6
383	40-1	"	"	Lt. Matte	2.02-1.27	1.6	2.5
384	20-1/2	"	"	Matte	1.81-1.15	1.6	2.6
385	60-6	20-1	30-15	Satin	1.52-0.91	1.7	2.5
386	40-4	"	"	Matte	1.75-0.98	1.8	3.2
387*	20-2	"	"	Dk. Matte	1.14-0.70	1.6	3.5
388	120-12	"	"	Brt Satin	1.20-0.83	1.5	3.2
389	120-2	"	30-90	Semi-Brt	1.89-1.04	1.8	2.8
390	60-1	"	"	Brt Satin	1.77-1.04	1.7	2.6
391	40-2/3	"	"	Matte	1.61-0.87	1.8	2.4

* Make-up additions of CrO_3
\+ Burnt edges
** Based on current differences between forward and reverse parts of cycle
Note: K= Kocour thickness measurement
M= Micrometer measurement at extreme edges and central area of specimen

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Edge-Ctr. (mil)	Ratio (K)	Ratio (M)
High Temperature (65°) PR Test Cont. (100 g/1 CrO_3)							
392	20-1/3	20-1	30-90	Dk. Matte	1.41-0.90	1.6	2.5
High Reverse							
393*	120-1-1/2	40-1	50-100	Semi-Brt	2.89-2.21	1.3	2.6
394	120-3	20-1	"	Brt Satin	2.44-2.17	1.1	1.5
395*	120-4	15-1	"	Satin	2.54-1.92	1.3	1.8
396	120-6	10-1	"	"	1.76-1.16	1.5	2.6
397	120-7-1/2	8-1	"	"	1.01-0.74	1.4	1.6
398*	120-6	10-1	"	"	1.27-0.99	1.4	1.8
399	120-12	5-1	"	Brt Satin	0.06-0.03	2.0	1.3
400*	120-10	6-1	"	Semi-Brt	0.05-0.05	1.0	1.0
Low Reverse							
401	120-12	20-1	50-25	Satin	2.48-2.25	1.1	1.1
402*	120-16	15-1	"	"	2.34-1.82	1.3	1.4
403	120-24	10-1	"	"	1.97-1.57	1.3	2.0
404*	60-6	20-1	"	Lt. Matte	2.30-1.59	1.4	1.6
405*	40-4	"	"	"	2.64-1.92	1.4	1.2
406	20-2	"	"	Matte	1.18-0.86	1.4	2.6
407*	120-6	40-1	"	Brt Satin	2.67-2.30	1.2	1.4
408	"	"	"	Semi-Brt	2.06-1.52	1.4	1.2
409*	60-3	"	"	Satin	3.05-2.57	1.3	1.1
410*	20-2	20-1	"	Lt. Matte	2.42-1.95	1.2	1.3
Equal Reverse							
411	20-1/2	40-1	50-50	Matte	2.34-1.95	1.2	1.5
412*	40-1	"	"	Lt. Matte	2.31-1.81	1.3	1.2
413	60-1-1/2	"	"	"	2.52-2.17	1.2	1.2
414*	180-4-1/2	"	"	Brt Satin	2.20-2.03	1.1	1.3
415	120-3	"	"	Semi-Brt	1.15-0.82	1.4	1.3
416*	120-6	20-1	"	Satin	1.99-1.35	1.5	1.4
417	60-3	"	"	"	2.24-2.05	1.1	1.1
418	40-2	"	"	Lt. Matte	2.66-2.01	1.3	1.2
Dilute bath + 25 g/1 NaOH							
419*	120-6	20-1	50-50	Blistered, no Cr at edges		--	--
420	no PR	--	50-	Brt Cr Ctr, edges bare		--	--
421*	"	--	30-	Brt$^+$	1.47-1.15	1.3	1.6
422	120-6	20-1	30-30	Satin$^+$	1.90-1.69	1.1	1.4

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Edge-Ctr. (mil)	Ratio (K)	Ratio (M)
New bath - 100 g/1 CrO_3							
423	no PR	--	30-	Brt	1.60-1.13	1.4	1.5
424	"	--	50-	Semi-Brt	2.54-2.06	1.2	1.3
425*	"	--	"	"	2.04-1.46	1.4	1.4
426	"	--	30-	"	1.56-0.98	1.6	1.9
Dilute bath + 10 g/1 NaOH							
427	no PR	--	50-	Semi-Brt	2.27-1.95	1.2	1.3
428*	"	--	"	Nr. Brt	1.96-1.65	1.2	1.3
429	"	--	30-	Brt	1.48-1.08	1.4	1.3
430	120-6	20-1	50-50	Lt. Matte	2.35-2.05	1.1	1.1
431	60-3	"	"	Matte	2.23-2.07	1.1	1.1
432*	120-3	40-1	"	Satin	2.55-2.08	1.2	1.1
433	180-4-1/2	"	.	Brt Satin	2.10-1.74	1.2	1.2
434*	40-1	"	"	Matte	2.60-2.16	1.2	1.2
435*	60-1-1/2	"	"	Lt. Matte	2.53-2.17	1.2	1.3
436	120-6	"	50-25	Brt Satin	2.12-1.62	1.3	1.3
437*	[illegible]	20-1	"	Matte	2.06-1.71	1.2	1.4
438	120-6	40-1	30-15	Satin	1.62-1.21	1.3	1.6
439*	120-1	"	30-90	"	1.55-1.24	1.3	1.2
440	60-1	20-1	"	Blk.	1.49-0.61	2.4	1.7
441*	"	"	"	"	1.67-0.61	2.7	3.3
442	120-3	40-1	30-30	Satin	1.96-1.55	1.3	1.5
443*	40-2	20-1	50-50	Matte$^+$	2.54-2.08	1.2	1.0
444	180-9	"	"	Satin	2.13-1.76	1.2	1.2
445	180-12	15-1	"	Brt Satin	1.85-1.48	1.3	1.1

* Make-up additions of CrO_3.

\+ Burnt edges.

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Ratio Edge-Ctr. (mil)	(K)	(M)
Dilute Bath (100 g/l CrO_3)							
446	120-6	20-1	50-50	Satin	2.68-1.91	1.4	2.2
448	120-3	40-1	"	"	2.64-2.22	1.2	1.2
449	No PR	--	50-	Nr. Brt	1.47-1.15	1.3	1.3
Dilute Bath + 5 g/l NaCl							
450	No PR	--	50-	Coarse Matte	2.26-1.63	1.4	1.6
451	120-3	40-1	50-50	Satin	3.14-2.56	1.2	1.3
10 g/l NaCl							
453	No PR	--	50-	Satin	2.58-2.25	1.2	1.0
454	120-3	40-1	50-50	Lt. Matte	2.80-2.36	1.2	1.4
455	120-6	20-1	"	Satin	2.16-1.94	1.1	1.0
15 g/l NaCl							
456	120-6	20-1	50-50	Coarse Matte	2.45-1.76	1.4	1.1
457	120-3	40-1	"	Brt Satin	2.02-1.84	1.1	1.0
458	No PR	--	50-	"	2.47-2.22	1.1	1.1
20 g/l NaCl							
459	No PR	--	50-	Brt	2.84-3.52	0.8	0.9
460	120-6	20-1	50-50	Cr Flaky	--	--	1.0
461	120-3	40-1	"	Satin	1.86-1.59	1.2	2.2
30 g/l NaCl							
462	No PR	--	50-	Grainy*	4.29-4.01	1.1	1.1
463	120-6	20-1	50-50	Flaked*	--	--	0.7
464	No PR	--	50-	Brt*	2.95-2.74	1.1	1.1
465	120-3	40-1	50-50	Ctr Brt*	0.64-2.24	0.3	0.6
466	120-6	20-1	"	Satin	2.09-1.73	1.2	1.1
High CO, Dilute Bath + NaCl							
467	120-6	20-1	100-100	Brt*	2.06-2.82	0.7	1.1
468	120-3	40-1	"	Satin	2.74-2.17	1.3	1.7
469	No PR	--	100-	Brt	3.05-2.60	1.2	1.5

Test No.	PR Cycle For.-Rev. (sec)	Effective Cycle**	Current Density For.-Rev. (asd)	Cr Surface	Cr Thickness Ratio Edge-Ctr. (mil)	(K)	(M)
High CO, Bath (Std. 250 g/l CrO_3)							
470	No PR	--	100-	Brt	2.26-1.70	1.3	5.0
471	120-6	20-1	100-100	Lt. Matte	1.89-1.34	1.4	1.9
472	120-3	40-1	"	Satin	2.39-1.71	1.4	2.0
473	"	"	150-150	Lt. Matte	2.53-1.80	1.4	2.7
474	120-6	20-1	"	Satin	2.48-2.03	1.2	2.3
475	No PR	--	150-	Brt	2.69-2.35	1.1	1.8
476	60-3	20-1	150-150	Lt. Matte	1.84-1.58	1.2	3.8
477	90-6	15-1	"	Satin	1.34-1.00	1.3	2.2
High CO, Standard Bath + 20 g/l NaOH							
478	120-6	20-1	150-150	Satin	2.59-2.34	1.1	1.1
479	120-3	40-1	75-75	"	2.26-1.88	1.2	1.8
480	"	"	150-150	Lt. Matte	2.25-1.77	1.3	2.6
481	No PR	--	150-	Satin	2.25-1.70	1.3	1.9
High Temperature + NaOH (10 g/l) (Dil. 100 g/l CrO_3)							
482	120-3	40-1	50-50	Satin	1.97-1.55	1.3	1.5
483	No PR	--	50-	Nr. Brt	2.26-1.79	1.3	1.6
484	120-6	20-1	50-50	Satin	1.76-1.70	1.0	1.6
485	60-3	20-1	"	"	1.97-1.49	1.3	1.2
486	120-3	80-1	50-25	Brt Satin	2.06-1.73	1.2	1.2
487	180-3	120-1	"	Semi-Brt	2.31-1.85	1.3	1.4
488	180-6	60-1	"	"	1.67-1.28	1.3	1.3
Dilute Bath + 10 g/l $NaCO_3$							
489	No PR	--	50-	Brt Satin	2.13-1.80	1.2	1.7
490	120-6	40-1	50-25	Satin	2.21-1.59	1.4	1.9
491	120-3	80-1	"	"	2.26-1.66	1.4	1.3
20 g/l $NaCO_3$							
492	120-3	80-1	50-25	Brt Satin	2.18-1.87	1.2	1.6
493	120-6	40-1	"	Satin	1.88-1.45	1.3	1.1
Dilute Bath + 10 g/l $NaCO_3$ + 10 g/l Na_2MO_4							
494	120-3	80-1	50-25	Ok. Matte	0.57-0.59	1.0	1.1
495	No PR	--	50-	"	1.48-1.15	1.3	2.4
496	120-6	40-1	50-25	Matte*	1.34-0.84	1.6	1.0
497	180-6	60-1	"	"	1.61-1.09	1.5	2.1

Test Deposits with Thickness Ratios less than 2.5

Test No.	Pulse on-off (millisec.)	Pulse Control (Volt-Amp.)	Base Level (% pulse level)	Temp-C.D. (°C/asd)	Cr Surface	Cr Thickness Ratio Edge-Ctr. (mils)
			Variable Pulse Test (cont.)			
549	50-10	V	25	65/45	Brt Satin	2.4
551	40-10	V	"	"	Satin	2.1
553	30-10	V	"	"	Matte	2.3
554	20-10	V	"	"	"	2.2
555	15-10	V	"	"	"	2.1
556	10-10	V	"	"	Satin	2.3
559	10-20	V	"	"	Matte	2.2
560	10-30	V	"	"	"	1.7
563	5-5	V	"	"	Satin	2.3
565	2-2	V	"	"	Lt. Matte	1.9
569	1-0.5	V	"	"	Brt Satin	2.3
571	0.5-0.2	V	"	"	Matte	2.2
572	100-0	-	100	"	Semi- Brt	2.3
573	"	-	"	55/45	Brt	2.3
575	3-2	V	25	"	Lt. Matte	2.4
576	3-1	V	"	"	Matte	2.1
580	20-10	V	"	"	"	2.3
582	50-50	V	"	"	Nr. Brt	2.1
583	30-30	V	"	"	Matte	2.0
586	30-50	A	"	"	"	2.1
587	10-10	A	"	"	Lt. Matte	2.0
588	5-5	A	"	"	Satin	2.4
			Base Voltage Level Test			
590	10-10	V	33	55/45	Matte	2.4
592	2-10	V	"	"	Nr. Brite	2.3
594	3-15	A	"	"	Brt	1.8

Test Deposits with Thickness Ratios less than 2.5

Test No.	Pulse on-off (millisec.)	Pulse Control (Volt-Amp.)	Base Level (% pulse level)	Temp-C.D. (°C/asd)	Cr Surface	Efficiency* (%)	Cr Thickness Ratio Edge-Ctr. (mils)
			Base Voltage Level Test				
595	4-20	V	33	55/45	Brt	11.9	1.5
597	5-15	V	"	"	Matte	7.0	1.9
598	7-21	V	"	"	Semi-Brt	9.8	2.3
599	7-21	V	40	"	Nr-Brt	8.1	1.8
600	7-21	V	45	"	Brt	9.5	2.4
601	7-71	V	50	"	"	12.3	2.4
651	7-21	V	60	"	"	13.0	1.7
603	7-21	V	75	"	"	14.4	1.4
604	7-21	V	26	"	Matte	6.4	2.3
652	7-21	V	33	"	Semi-Brt	6.9	2.0
660	7-21	V	75	"	Brt	14.0	1.9
659	7-21	V	40	"	"	9.7	2.0
657	7-21	V	50	"	Semi-Brt	11.9	2.2
			Pulse Ratio Test				
607	10-30	V	33	55/45	Matte	6.1	2.0
608	12-36	V	"	"	"	9.8	2.2
653	16-48	V	"	"	Satin	11.8	2.0
613	20-60	V	"	"	Matte	10.0	1.6
614	30-60	V	"	"	Satin	8.7	2.1
615	50-60	V	"	"	"	8.4	2.0
654	50-50	V	"	"	Semi-Brt	10.1	1.8
619	10-50	V	"	"	Lt. Matte	10.5	1.7
620	10-60	V	"	"	Semi-Brt	6.5	2.1
621	8-48	V	"	"	Nr-Brt	11.9	2.1
622	15-75	V	"	"	Lt. Matte	9.4	2.2
654	50-50	V	"	"	Semi-Brt	9.9	1.8
624	9-36	V	"	"	Satin	7.2	2.2
625	3-15	V	"	"	Brt	8.1	2.1
626	100-0	-	"	"	"	9.9	2.1

Test Deposits with Thickness Ratios less than 2.5 (cont'd.)

Test No.	Pulse on-off (millisec.)	Pulse Control (Volt-Amp.)	Base Level (% pulse level)	Temp-C.O. (°C/asd)	Cr Surface	Efficiency* (%)	Cr Thickness Ratio Edge-Ctr. (mils)
			Pulse Ratio Test (cont'd.)				
628	15-100	V	"	"	Semi-Brt	9.6	2.1
629	25-100	V	"	"	Brt-Satin	10.5	1.7
656	10-30	V	50	"	Brt	12.4	2.2
631	15-75	V	"	"	Brt-Satin	12.4	2.2
632	12-60	V	"	"	Nr-Brt	13.5	2.1
633	10-50	V	"	"	Brt	11.7	2.0
634	10-40	V	"	"	"	11.2	2.0
635	10-20	V	"	"	"	9.9	2.2
636	10-10	V	"	"	Nr-Brt	11.4	2.2
637	10-5	V	"	"	Brt	11.8	1.7
638	10-33	V	"	"	"	10.9	1.9
639	15-15	V	"	"	Nr-Brt	14.1	1.7
641	25-5	V	"	"	Brt	12.2	2.2
642	50-5	V	"	"	"	12.2	2.2
643	100-5	V	"	"	"	14.5	2.2
644	20-60	V	"	"	Semi-Brt	10.9	2.0
645	9-36	V	"	"	Brt	11.7	2.2
646	50-60	V	"	"	Brt-Satin	13.0	2.2
647	30-60	V	"	"	Semi-Brt	12.8	2.0
648	12-48	V	"	"	Brt	12.8	1.8
650	12-36	V	"	"	Nr-Brt	9.2	2.0
			Duplicates of Low Ratio Deposits				
658	3-15	V	33	55/45	Brt	10.3	2.0
661	20-60	V	"	"	Satin	9.9	2.1
662	10-50	V	"	"	Nr-Brt	11.2	1.8
663	10-5	V	"	"	Brt	11.0	1.7
664	15-15	V	"	"	Nr-Brt	11.1	1.9

Test Deposits with Thickness Ratios less than 2.5 (cont'd.)

Test No.	Pulse on-off (millisec.)	Pulse Control (Volt-Amp.)	Base Level (% pulse level)	Temp-C.O. (°C/asd)	Cr Surface	Efficiency* (%)	Cr Thickness Ratio Edge-Ctr. (mils)
			Duplicates of Low Ratio Deposits (cont'd.)				
665	10-30	V	25	"	Matte	8.6	2.0
666	25-100	V	50	"	Nr-Brt	11.5	1.8
667	4-20	V	33	"	Brt	9.7	2.3
668	15-15	V	50	"	Nr-Brt	11.4	1.8
669	10-5	V	"	"	"	11.0	2.0

* The deposition efficiency is determined from the thickness of the Cr near the center of the panel, and the value is thus somewhat affected by the distribution of the deposit.

Test No.	Pulse on-off (millisec.)	Pulse Control (Volt-Amp.)	Base Level (% pulse level)	Temp-C.D. (°C/asd)	Cr Surface	Efficiency %	Cr Thickness Ratio Edge-Ctr.
Duplication of Low Ratio Deposits (cont.)							
670	4-20	V	33	55/45	Semi-brt.	9.3	2.2
671	4-20	V	"	"	Brt.	10.6	2.4
672	25-75	V	50	"	Brt.	13.2	1.9
673	100-0	-	-	"	Brt.	11.7	2.5
674	16-48	V	33	"	Satin	9.3	1.7
675	16-48	A	"	"	Semi-brt.	13.9	1.8
Dilute Bath - (100 g/l CrO_3)							
676	16-48	V	33	55/45	Nr. brt.	12.7	1.4
677	25-100	V	"	"	Brt.	11.8	1.3
678	15-15	V	50	"	Lt. matte	13.7	1.8
679	100-0	-	-	"	Brt.	14.9	1.6
680	10-5	V	50	"	"	12.2	1.3
681	3-15	V	33	"	"	9.7	0.9
682	50-50	V	"	"	"	12.4	1.2
683	7-21	V	75	"	"	13.4	1.2
684	10-30	V	25	65/45	Nr. brt.	7.0	1.5
Concentrated Bath - (500 g/l CrO_3)							
685	3-15	V	33	55/45	matte	7.3	3.9
686	50-50	V	"	"	"	11.4	2.8
687	7-21	V	75	"	Nr. brt.	7.0	5.6
688	10-5	V	50	"	matte	12.2	3.3
689	25-100	V	33	"	"	8.5	3.8
690	16-48	V	"	"	"	8.1	2.7
691	100-0	-	-	"	Nr. brt.	6.8	7.4
692	10-30	V	25	"	Satin	12.9	1.6

Test No.	Pulse on-off (millisec.)	Pulse Control (Volt-Amp.)	Base Level (% pulse level)	Temp-C.D. (°C/asd)	Cr Surface	Efficiency %	Cr Thickness Ratio Edge-Ctr.
Current Controlled Pulse							
693	3-15	A	33	55/45	Brt.	9.5	2.0
694	15-15	A	50	"	Brt.	14.0	2.4
695	25-100	A	33	"	"	11.6	2.0

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Unclassified report.

This report covers an investigation of the effects of periodic reverse current and pulsed direct current on properties and metal distribution of chromium electrodeposits. The plating current was reversed for short periods of various lengths during deposition of chromium at various current densities and in baths at several concentrations of chromium trioxide or CrO_3.

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