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Rock Island Arsenal Laboratory



TECHNICAL REPORT

LAMINAR CHROMIUM ELECTRODEPOSITS

By

Jodie Doss

Department of the Army Project No. 593-32-006

Ordnance Management Structure Code No. 5010.11.810

Report No. 61-3826

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Approved by:


A. C. HANSON
Laboratory Director

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Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

Steel specimens were laminar (discrete layers or bands) chromium plated and adhesion tested by the high speed rotor technique. Four different pretreatments of the steel prior to plating were evaluated as to their effect on the adhesion of the plate to the basis metal. Two plating methods were utilized to produce laminar deposits. Corrosion tests were performed on laminar and nonlaminar chromium plated specimens. The laminar chromium adhesion was not as great as the non-laminar chromium. The oxalic acid etched steel and the electropolished pretreated steel had the best chromium adhesions, with the oxalic acid etched steel slightly greater than the electropolished pretreated steel. The laminar chromium electrodeposits had better corrosion resistance than the nonlaminar electrodeposits of the same thickness.

RECOMMENDATIONS

Laminar chromium plating should be further investigated to determine corrosion resistance of thicker deposits containing various numbers of laminae and laminae of a wider range of thicknesses.

LAMINAR CHROMIUM ELECTRODEPOSITS

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LAMINAR CHROMIUM ELECTRODEPOSITS

OBJECT

To investigate the adhesion and corrosion resistance of laminar chromium electrodeposits.

INTRODUCTION

Chromium plating has been a commercial process since the early twenties.

A chromium electrodeposit has certain properties that make it an ideal coating for steel such as extreme hardness and permanent luster. Still the corrosion resistance of chromium deposits leave much to be desired. Very thin coatings have a number of pores that decrease in number as the thickness of the plate increases. Meanwhile, as the thickness of the plate increases, the structure becomes pitted and cracked as shown in Figure 1. The pores and cracks in the chromium plate allow corrosion media to penetrate the plate and attack the basis metal⁽¹⁾.



FIGURE 1

TYPICAL CHROMIUM PLATE CRACK PATTERN

Orig. Mag. - 50

Neg. No. 8638

Another difficulty encountered in chromium deposits is lamination. A laminar chromium electrodeposit contains discrete layers or bands within the plating structure. Laminar chromium electrodeposits have been observed since the advent of chromium plating. It was first discovered in chromium electrodeposits on steel that had been removed from the plating solution for measurements prior to final plating. It was also found at the interface of new chromium plating over old chromium plating or in chromium iron alloy plating⁽²⁾.

The production of laminar plating has been discouraged because it was believed to produce coatings of inferior adherence. The reason for the poor adhesion was believed to be due to a passive film of chromium oxide on the surface. It has been found that laminar chromium plating through a cathode film formed between successive layers of plating, produced laminae which bond much more strongly to one another than those produced by the usual technique of anodic etching between individual layers⁽³⁾.

The purpose of this investigation was to determine if laminar chromium electrodeposits have poor adhesion. Also, the purpose was to determine if laminar chromium electrodeposits would have less porosity and better corrosion resistance.

PROCEDURE AND RESULTS

A literature review for methods to test adhesion revealed the high speed rotor technique, which it was believed, would give the most accurate results⁽⁴⁾. Springfield Armory, the technical supervisor of investigations being performed at the University of Virginia⁽⁵⁾, cooperated in this investigation.

Rock Island Arsenal, through Springfield Armory, obtained from the Department of Physics, University of Virginia, sixteen one-eighth inch diameter cylindrical heat treated and ground rods of FS 4140 steel. The rods were 3.5" long with a cylindrical slot approximately 1/16" from one end.

Sets of two steel rods were subjected to four different pretreatments prior to chromium plating.

The first two rods were designated S-1 and S-2. They were anodically etched in a 70 percent sulfuric acid solution for two minutes at a current density of 3 amps/in².

The next two rods were designated O-1 and O-2. They were anodically etched in a ten percent oxalic acid solution for three minutes at a current density of 2 amps/in². An

oxalic etch was reported to be one of the best etchants for steel prior to chromium plating⁽⁶⁾.

The third group of two rods was designated E-1 and E-2. They were electropolished in a 50-50 mixture of concentrated sulfuric acid and concentrated phosphoric acid solution for two minutes at a current density of 3 amps/in².

The fourth group was designated D-1 and D-2. They were alternately anodically (5 sec.) and cathodically (10 sec.) cleaned in an alkaline cyanide solution for five minutes.

The eight rods were placed, by means of eight set screws, in a specially prepared plating rack. This rack had eight anodes made of perforated 2.5 inch diameter steel tubes which were welded together. This anode apparatus was lead-tin plated. The eight rods were held in the center of each anode tube by means of the set screw that projected through a plastisol covered copper fixture. The eight steel rods were seated on the bottom of the plastisol covered cathode fixture.

The racked eight rods were reverse etched in a sulfate free 25 percent chromic acid solution for five minutes. The racked rods were then moved to the chromium plating solution and reverse etched for thirty seconds prior to chromium plating. The chromium plating solution was a conventional 33 oz. CrO₃ and 0.33 oz. of sulfate per gal. The steel rods were plated at 5 amperes per square inch for 16 hours.

It had recently been observed by the author that chromium electrodeposits could be laminated if the temperature of the plating solution was alternated between 124°F to 138°F. There was a possibility that current fluctuations could be producing the laminar coatings. However, this was disproven by the use of a recordomatic watt meter. The wattage remained constant during the plating of the laminar coatings. Therefore, the laminar coatings were produced by alternating the temperature of the plating solution. The above eight rods were then chromium plated in this manner.

In order to reveal lamination in the chromium deposit it was necessary to cross section the rod and etch the chromium deposit. Figure 2 is a photomicrograph of a plated cross sectioned rod after a two percent nital etch (HNO₃ + alcohol). There was no visible evidence of lamination of the deposit. Figure 3 is a photomicrograph of the same cross sectioned rod after a concentrated hydrochloric acid etch. This photomicrograph revealed a laminar chromium deposit. The nital solution did not etch the chromium deposit. The hydrochloric acid solution did etch the chromium deposit and revealed the laminar structure.



Bakelite
Mount

Chromium
Plate

Steel
Rod

FIGURE 2

Neg. #1625

Nital Etched



Bakelite
Mount

Chromium
Plate

Steel
Rod

FIGURE 3

Neg. #1626

Concentrated Hydrochloric Acid Etched

CROSS SECTIONS OF ETCHED LAMINAR CHROMIUM PLATED RODS
100X .009 in. Plate

The chromium plated rods were forwarded through Springfield Armory to the University of Virginia for adhesion tests. The results of the tests are presented in Table I. The chromium plated rods that had been oxalic acid etched had the highest average adhesion values. The chromium plated steel specimens that had been sulfuric acid etched had the second highest average adhesion values. The chromium plated steel specimens that had been alkaline cyanide cleaned were next. The chromium plated steel specimens that had been electropolished gave the lowest average adhesion values.

The O-2 and E-1 chromium plated rods were cross sectioned and photomicrographed at the University of Virginia. The coating structure of the O-2 rod is shown in Figure 4. The coating structure of the E-1 rod is shown in Figure 5. Figure 6 is a photomicrograph of a cross sectioned nonlaminar chromium plated rod that was plated at the University of Virginia.

A second group of eight rods were given the same pretreatments and chromium plated in the same manner so as to produce laminar coatings. The specimens were plated for 16 hours at a current density of 2ASI. This second group of chromium plated rods were forwarded through Springfield Armory to the University of Virginia for adhesion tests. The results of the second tests are shown in Table II.

The results of the tests indicated that the bond strength of the chromium plated steel rods that had been alkaline cyanide or electropolished pretreated increased over the previous tested specimens. The electropolished steel rods that had been chromium plated in the second test group had the greatest chromium-steel bond strength of all the test specimens. The bond strength of the chromium plated steel rods that had been oxalic acid etched decreased from test group one to test group two.

Other techniques along with the one already utilized were explored in order to produce thinner laminar chromium electrodeposits. The plating methods investigated were:

1. Plating for 15 minute intervals and stop plating for one minute intervals. The solution was maintained at $131 \pm 2^{\circ}\text{F}$.

2. Plating for 15 minute intervals and stop plating with the removal of the plating specimen from the plating solution for one minute intervals. The solution was maintained at $131 \pm 2^{\circ}\text{F}$.

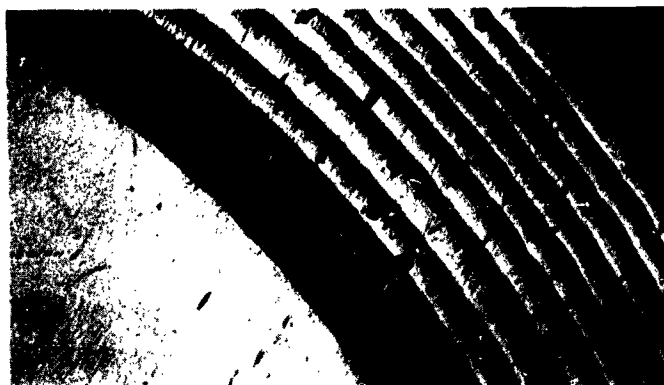
TABLE I⁽⁷⁾CHROMIUM PLATED AT ROCK ISLAND ARSENAL

<u>PLATING NO.</u>	<u>FILM THICKNESS (INCHES)</u>	<u>MAX. SPEED RPS</u>	<u>MAX. STRESS PSI</u>	<u>AVG. STRESS PSI</u>	<u>PRE- TREATMENTS AVERAGE STRESS PSI</u>
Electrolytically Alkaline-Cyanide Cleaned					
D-1	0.0208	28,600	35,930	35,370	31,070
	0.0221	27,800	36,690		
	0.0213	27,750	34,870		
	0.0221	26,750	33,970		
D-2	0.0264	22,080	29,310	26,770	
	0.0269	18,130	20,260		
	0.0271	19,250	23,060		
	0.0281	22,950	34,440		
Electropolished					
E-1	0.0272	22,480	31,470	23,050	25,885
	0.0273	18,480	21,370		
	0.0285	21,100	29,550		
	0.0282	12,250	9,810		
E-2	0.0265	23,100	32,140	28,720	
	0.0275	23,450	34,830		
	0.0282	18,880	23,360		
	0.0279	19,500	24,560		
Electrolytically Oxalic Acid Etched					
O-1	0.0257	23,450	31,810	30,770	38,145
	0.0263	23,750	33,660		
	0.0265	23,120	32,250		
	0.0266	20,450	25,360		
O-2	0.0191	34,150	45,780	45,520	
	0.0202	35,380	52,800		
	0.0201	32,550	44,390		
	0.0204	30,250	39,090		

TABLE I (Cont.)

<u>PLATING NO.</u>	<u>FILM THICKNESS (INCHES)</u>	<u>MAX. SPEED RPS</u>	<u>MAX. STRESS PSI</u>	<u>AVG. STRESS PSI</u>	<u>PRE- TREATMENTS AVERAGE STRESS PSI</u>
Electrolytically Sulfuric Acid Etched					
S-1	0.0262	21,650	27,820		
	0.0267	17,900	19,500	26,730	
	0.0274	21,350	28,760		
	0.0280	21,780	30,820		
					32,375
S-2	0.0165	32,800	35,150		
	0.0177	32,140	36,850	33,020	
	0.0173	32,850	36,940		
	0.0177	34,730	43,150		

Steel
Rod



Bakelite
Mount

Chromium
Plate

FIGURE 4 0-2 Oxalic Acid Etched .020 in. Plate



FIGURE 5 E-1 Electropolished .028 in. Plate



FIGURE 6 Nonlaminar Chromium Plate .027 in. Plate
Plated at Univ. of Virginia

CROSS SECTIONS OF CHROMIUM PLATED STEEL RODS
PHOTOMICROGRAPHED AND ADHESION TESTED
AT THE UNIVERSITY OF VIRGINIA 100X

TABLE II(8)

ADHESION OF CHROMIUM DEPOSITED BY ROCK ISLAND ARSENAL

PLATING NO.	FILM THICKNESS (INCHES)	MAX. SPEED RPS	MAX. STRESS PSI	AVERAGE STRESS PSI	PRE- TREATMENTS AVERAGE STRESS PSI	REMARKS
Electrolytically Alkaline Cyanide Cleaned						
D-3	0.0169	29,380	29,160			
	0.0173	32,650	37,100	32,160		
	0.0172	27,170	25,520			
	0.0172	33,450	38,680			
D-4	0.0055	66,650	41,340		37,640	Steel Rotor ex- panded slightly before bond failure.
	0.0059	65,260	42,770	43,120		
	0.0062	64,150	43,630			
	0.0064	63,850	44,750			
Electropolished						
E-3	0.0064	58,880	37,840			
	0.0063	61,280	40,250			
	0.0067	59,700	40,270	39,990		
	0.0067	60,180	41,600		40,420	
E-4	0.0143	39,350	42,520			
	0.0139	40,970	44,550	40,860		
	0.0144	33,830	31,690			
	0.0146	39,850	44,710			

TABLE II (Cont.)

<u>PLATING NO.</u>	<u>FILM THICKNESS (INCHES)</u>	<u>MAX. SPEED RPS</u>	<u>MAX. STRESS PSI</u>	<u>AVERAGE STRESS PSI</u>	<u>PRE- TREATMENTS AVERAGE STRESS PSI</u>	<u>REMARKS</u>
Electrolytically Oxalic Acid Etched						
O-3	0.0188	31,770	38,930			
	0.0189	31,370	38,210			
	0.0189	31,400	38,310	38,800		
	0.0192	31,680	39,780			
O-4	0.0212	28,270	35,910		36,525	
	0.0210	28,780	36,770			
	0.0210	28,850	36,970	34,250		
	0.0210	24,830	27,380			
Electrolytically Sulfuric Acid Etched						
S-3	0.0182	29,050	31,190			
	0.0175	29,900	31,480			
	0.0183	30,950	35,690	34,350		
	0.0185	32,160	39,070			
S-4	0.0239	18,300	17,610		28,490	
	0.0238	24,380	31,080			
	0.0242	19,130	19,560	22,630		
	0.0241	20,470	22,270			

3. Conventional plating while maintaining the plating solution at 131 ± 2 degrees F. for the entire plating cycle.

4. Reversing the current for 15 seconds after every 15 minutes of plating. The solution was maintained at 131 ± 2 degrees F.

5. Plating while regulating the temperature alternately between 124°F. to 138°F.

The chromium plated specimens were sectioned and photomicrographs made of the cross sectioned areas. Laminar chromium electrodeposits were observed in chromium deposits produced by Method 2 and Method 5. Laminar chromium electrodeposits were not found in the deposits produced by the other three plating methods.

Plating thicknesses of from one to three mils (.001 to .003 in.) were to be utilized for the corrosion resistance tests. Preliminary experiments were conducted to ascertain which plating method should be used in order to produce the laminar coatings. Two $1/8$ " dia. steel rods were laminar chromium plated with 1.1 mils of chromium. Rod No. 1 had a chromium deposit in which there were only three laminae. It was produced by alternating the temperature of the plating solution between 124°F. to 138°F. (Method 5). A photomicrograph of the cross sectioned plated rod is shown in Figure 7. The No. 2 steel rod was laminar chromium plated by stopping the plating and removing the rod from the plating solution for one minute intervals during the plating cycle (Method 2). The chromium deposit had five laminae. A photomicrograph of the cross sectioned chromium plated rod is shown in Figure 8. Figure 9 is a photomicrograph of a cross sectioned four laminae 1.7 mil thick chromium electrodeposit. This specimen was laminar chromium plated using the same procedure used for the latter specimen. Because of the versatility of this method it was used to laminar chromium plate the corrosion test specimens.

Eight $2" \times 3" \times 1/8"$ FS 1020 surface ground steel panels were vapor degreased and their thicknesses measured. Four of the panels were chromium plated for 2.5 hours at a current density of approximately 1.3 ASI. The other four panels were plated for 2 hours and 35 minutes. After every thirty minutes of plating, the plating was stopped and the panels were removed from the plating solution for one minute intervals in order to produce five laminar deposits. The plated panels were again measured and the thickness of plating calculated. The average thickness of plating on the eight panels was 1.3 mils. There were manifestations of poor adhesion on the edges



FIGURE 7 Laminated By Plating Solution Neg. #3083
Temperature Changes

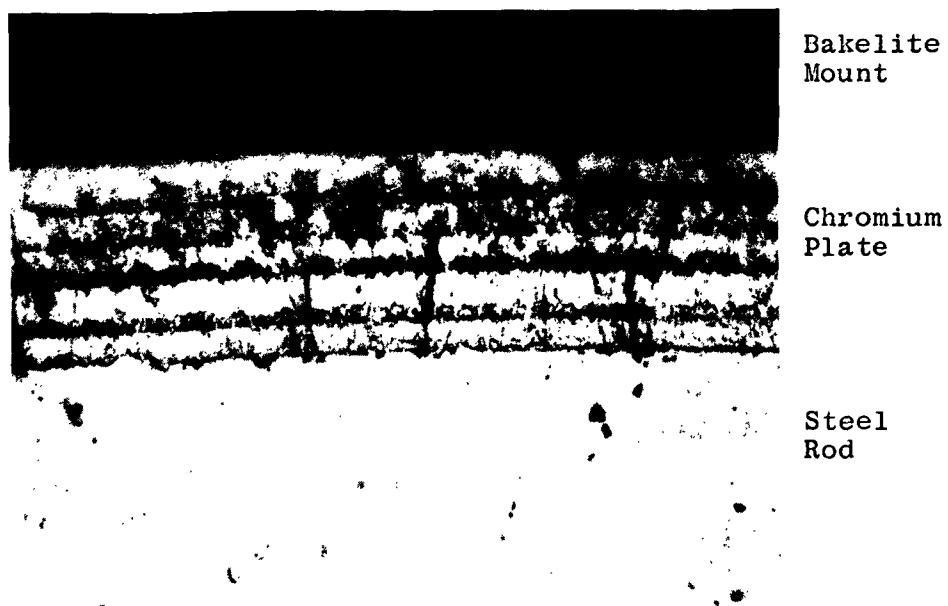


FIGURE 8 Laminated By Periodic Removal Neg. #3106A
From Plating Solution

CROSS SECTIONS OF LAMINAR CHROMIUM PLATED STEEL RODS
.0011 in. Plate 1000X

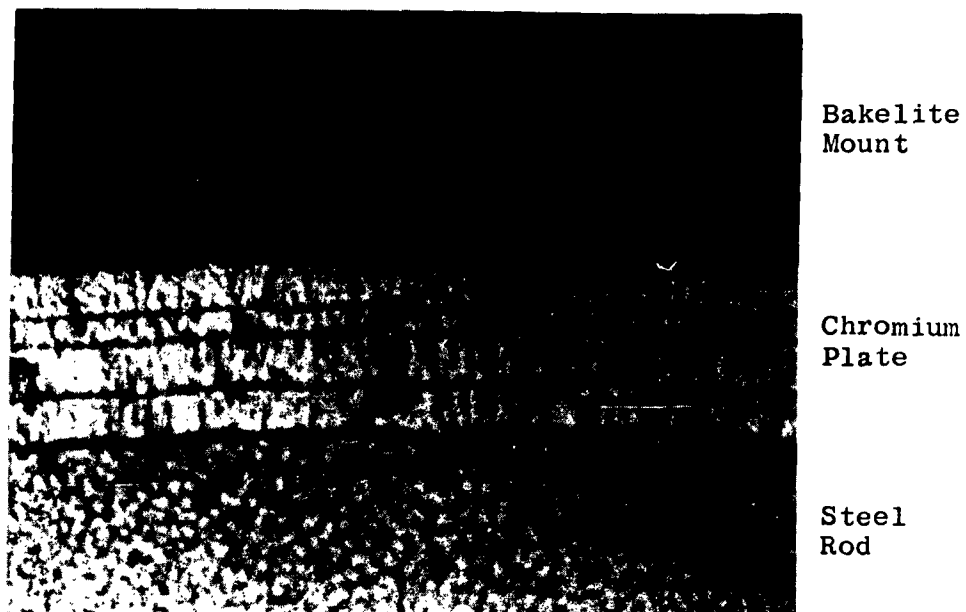


FIGURE 9 Laminated By Periodic Removal Neg. #3118
 From Plating Solution

CROSS SECTION OF LAMINAR CHROMIUM PLATED STEEL ROD
.0017 in. Plate 1000X

and corners of the 2" x 3" steel panels. These areas were, of course, high current density areas during the plating operation. It is apparent therefore that high current density areas must be avoided in the formation of laminar electrodeposits.

The corners and edges of the eight plated panels were covered with wax. The panels were salt fog tested in a salt spray cabinet containing a neutral five percent salt solution. The salt spray cabinet was operated in accordance with Method 811.1 of Federal Test Method Standard No. 151. After twenty-four hours in the salt fog environment, the nonlaminar chromium plated specimens had red corrosion products present over the face of the specimens. The laminar chromium plated specimen had only a small amount of corrosion present.

Two more groups of test specimens were corrosion tested in the salt spray cabinet. The first group of specimens had a plating thickness of 2.5 mils, one half of this group contained five laminae coatings. The other half of the group had nonlaminar coatings.

The second group of laminar and nonlaminar plated specimens had a plating thickness of 1.7 mils. The laminar coatings containing seven instead of five laminae. The purpose of this was to ascertain if seven laminae coatings would be more corrosion resistant than five laminae coatings.

The laminar chromium electrodeposits were more corrosion resistant than the nonlaminar chromium electrodeposits. The seven laminae coatings were not more corrosion resistant than the five laminae coatings.

DISCUSSION

The high speed rotor technique employs a coated steel rotor that is magnetically freely suspended. The rotor is spun inside a vacuum chamber by a rotating magnetic field in a manner similar to that of the armature of an induction or synchronous motor. The rotor speed seems to be limited only by the strength of the rotor. Speeds of 1×10^6 RPS have been obtained. In a measurement of adhesion the speed of the chromium plated rotor is increased until the centrifugal force exceeds the bond strength of the deposit to the basis metal resulting in removal of the deposit. It is then possible to calculate the maximum force necessary to remove the chromium plating from the steel⁽⁴⁾. A complete description of the apparatus and calculations can be found in a report by Dancy and Kuhlthau⁽⁹⁾.

The results of the adhesion tests presented in Tables I and II revealed very erratic information. While attempting to correlate the results it was noticed that the average stress was depending upon the thickness of the plate. A thin coating required a larger force to remove the plate than a thick coating. In Table III an attempt was made to group the four different pretreated chromium plated specimens according to thickness. The first group in Table III had an average thickness of approximately .027 in. The chromium plated steel specimen that had been oxalic acid etched had the highest stress value although the average plate thickness is a little less than the rest of group. The chromium plated steel specimens that had been alkaline cyanide cleaned and sulfuric acid etched were next with the same average plate thickness and approximately the same stress values. Both chromium plated specimens that had been electropolished had slightly thicker plates than the rest of the specimens of the group. Yet, the chromium plate on one of the specimens had a higher average stress value and the other had a lower average stress than the chromium plated specimens that had been alkaline cleaned and sulfuric acid etched.

In Group 2 of Table III the average thickness of plate was approximately .017 in. The electropolished chromium plated specimen had the highest adhesion value but also the lowest film thickness. The chromium plated specimen that had been oxalic acid etched had the highest film thickness of the group, but had an adhesion value very close to the chromium plated specimen that had been electropolished. The sulfuric acid etched specimens had a thicker plate than the alkaline cyanide cleaned specimens but also had a higher adhesion value.

In Group 3, of Table III, the averages of Group 1 and 2 were combined and averaged. This gave an average plating thickness with a smaller deviation of each specimen from the mean. The average stress values indicated that the sulfuric acid etched steel-chromium plate had better adhesion than the alkaline cleaned steel-chromium plate. The electropolished steel-chromium plate had a greater adhesion than both of the above pretreated steel-chromium plates. The oxalic acid etched steel-chromium plate had the greatest adhesion of all the specimens. The electropolished steel-chromium plating did have a slightly thicker plate than the oxalic acid etched steel-chromium plated specimen. This would lead one to predict that if the plate were .001 in. thinner the electropolished steel-chromium plate would have a larger average stress, on the other hand the average thickness of plate for E-4 in Table II is .014 in. This gave an average stress of 40,860 psi. The average thickness of plate for 0-2 in Table I was .020 in. This gave a larger average stress of 45,520 psi.

TABLE III

ADHESION OF CHROMIUM DEPOSITED AT ROCK ISLAND ARSENAL
SIMILAR THICKNESS OF PLATE VERSUS AVERAGE STRESS

<u>PLATING NUMBER</u>	<u>AVERAGE THICKNESS (INCHES)</u>	<u>AVERAGE STRESS (PSI)</u>
<u>Group 1</u>		
D-2	.0271	26,770
E-1	.0278	23,050
E-2	.0275	28,720
O-1	.0263	30,770
S-1	.0271	26,730
} Avg. .0277		
} Avg. 25,885		
<u>Group 2</u>		
D-3	.0172	32,160
E-4	.0143	40,860
O-3	.0184	38,800
S-3	.0181	34,350
<u>Group 3</u>		
D-2 + D-3	.0222	27,465
E-1 + E-2 + E-4	.0232	33,473
O-1 + O-3	.0224	34,785
S-1 + S-3	.0226	30,540

Pretreatments code:

D = Alkaline-cyanide cleaned
E = Electropolished
O = Oxalic acid etched
S = Sulfuric acid etched

The average stress values for all the laminar chromium plate were somewhat lower than the nonlaminar chromium plate that had been plated and tested at the University of Virginia. The average stress values for all the laminar chromium plate were still not as low as had been expected.

The time necessary to heat the plating solution to 138°F. and allow the solution to cool to 124°F. was too long a time cycle to have enough laminae in a one mil (.001 in.) thick coating. Figure 7 illustrates such a plate. There are only three laminae in the plate. It has been observed that in most instances at least three laminae are needed in order to stop a flaw or void in the plate. Close study of photomicrographs in this report bears out this statement.

The other plating method utilized (stopping the plating with the removal of the specimen from the plating solution for one minute intervals) to produce laminar coatings was more practical. It was possible to produce more laminae in a thin plating by the latter method than by the temperature cycling method. Figure 8 reveals five laminae in the coating. The five laminae platings exhibited better corrosion resistance than the nonlaminar platings. The plating containing seven laminae did not indicate better corrosion resistance to salt fog than the deposit containing five laminae.

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LIST OF PRIOR REPORTS

<u>R.I.A. Lab. No.</u>	<u>Date Issued</u>	<u>Title</u>
43-3682-1	9-2-43	Performance Test of R975-EC-1 Engine Equipped With Porous Chromed Cylinder, Plated by United Chromium Co., Detroit
43-3682	9-13-43	Experimental Reclamation of General Motors Corporation Diesel Engine (Model 6046) Crankshaft By Hard Chrome Plating The Bearing Journals
43-3682-4	10-10-44	Investigation of Reclamation of Continental-Wright R975-C1 Engines By Porous Chrome Plating The Crank- shafts and Cylinder Barrels
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RIA Lab. Rep. 61-3826, 19 Oct 61, 23 p., incl.
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Steel specimens were laminar (discrete layers
or bands) chromium plated and adhesion tested
by the high speed rotor technique. Four
different pretreatments of the steel prior to
plating were evaluated as to their effect on
the adhesion of the plate to the basis metal.
Two plating methods were utilized to produce
laminar deposits. Corrosion tests were per-
formed on laminar and nonlaminar chromium
plated specimens. The laminar chromium ad-
hesion was not as great as the nonlaminar
chromium. The oxalic acid etched steel and
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