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FINAL TECHNICAL REPORT

"EVALUATION OF PRINCIPAL FACTORS FOR OBTAINING DUCTILE HOT-HARD CHROMIUM ELECTROPLATE FOR CANNON"

O.O. Project Number: TR3-3003B

W.A.L. File Number: 691.1/19-13

Contract: W33-019 ORD 6319

Classification: ~~RESTRICTED~~

September 1, 1948

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October 28, 1948

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Attention of Laboratory

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Contract W33-019-ord-6319
W.A.L. File No. 691.1/19-13
O.O. Project No. TR3-3003B

In accordance with paragraph 5 of the "Memorandum on Reports in Connection with Contracts under the Technical Supervision of Watertown Arsenal Laboratory", letter of February 24, 1948, from Commodity Section, ORDEE No. 160/6319, a copy of the Final Report, dated September 1, 1948, on the reference contract, is enclosed for approval. All other copies will be held for distribution until advised of approval by the Cleveland Ordnance District.

Very truly yours,

Charles L. Faust

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FINAL TECHNICAL REPORT

Contractor: Battelle Memorial Institute

Agency: Office, Chief of Ordnance, ORDTR - Cannon

Ordnance District: Cleveland, Ohio

Contract Number: W33-019 ORD-6319 W. A. L. File No. 691.1/19-13

O.O. Project Number: TR3-3C03B

Priority: War Department 2B

Title of Project: "Evaluation of Principal Factors for Obtaining Ductile Hot-Hard Chromium Electroplate for Cannon."

Authors: Harry F. Ross, William C. Schickner, Cloyd A. Snavely, and Charles L. Faust.

Object: To establish fundamental information leading to the development of an erosion-resistant chromium or chromium alloy electroplate for lining gun tubes.

Summary: Results of experiments are reported dealing with physical characteristics of chromium electroplate and with new electrolytes for the electrodeposition of chromium. The new electrolytes fall in the three classes: fused salt baths, aqueous baths, and organic type baths. The new information serves as the basis for discussion of the possibilities for producing a hot-hard electrocoating for cannon.

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Conclusions

Experimental data show that nitriding or boronizing of chromium plate is not a practicable method for counteracting the softening normally occurring when the plate is heated above the recrystallization temperature. A considerable amount of work with various electrolytes has not indicated a method for producing ductile chromium plate. The best possibilities for a hot-hard and ductile electroplate appear to lie in the alloy plating field.

Report Period

This report covers the period from September 19, 1947, to September 1, 1948.

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FINAL TECHNICAL REPORT

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EVALUATION OF PRINCIPAL FACTORS FOR OBTAINING DUCTILE, HOT-HARD CHROMIUM ELECTROPLATE FOR CANNON*

by

Harry F. Ross, William C. Schickner,
Cloyd A. Snavely, and Charles L. Faust

SCOPE OF WORK

The work herein reported falls under the following general headings:

1. An evaluation of the known factors governing the ductility and hot-hard properties of electrodeposited chromium metal.

2. A study of the cathode film constituents and their effect on film pH during chromium plating.

3. A survey of the possibilities for improving the hot hardness of substantially stress-free, low-contraction chromium plate as by deposition of chromium alloy, or by special hardening treatments.

4. Investigations directed toward development of an improved chromium plating process to produce ductile chromium plate.

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* The experimental data obtained in this work are recorded in Laboratory Record Books No. 2988, pages 72-100, and No. 3561, pages 1-87.

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INTRODUCTION

The work reported herein is divided into two main phases. In the first phase the studies were related to the physical characteristics of chromium plate. The information thus gained was used as the basis for the second phase of the work which involved attempts to produce chromium plate with improved physical properties.

EVALUATION OF KNOWN FACTORS GOVERNING THE DUCTILITY AND HOT-HARD PROPERTIES OF ELECTRODEPOSITED CHROMIUM METAL

The factors governing the physical characteristics of chromium plate were discussed in some detail in the final report on a previous contract(1). The conclusions drawn therein were based on limited experimental data. In the present work, new data were obtained which are believed to provide positive support for the earlier conclusions. In addition, new information was gained which was not predicted by the earlier work.

The known factors relating to the hardness and ductility of chromium plate may be listed as follows:

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(1) Final report submitted July 28, 1947, by Battelle Memorial Institute under Army Ordnance Contract W33-019 ORD-6267.

Grain size

Internal stress

Occluded hydrogen

Dispersed oxide

Metallographic studies of the structures of "as plated" and annealed chromium plates revealed the annealing effects illustrated in Figures 1, 2, and 3. Recrystallization and grain growth did not take place at 300°C., yet are clearly evident after treatment at 500°C. The occurrence of recrystallization and grain growth at treatment temperatures between 300°C. and 500°C. is thus established as an observed fact instead of a postulated theoretical deduction from other facts. Additional information relevant to these conclusions, but not essential to their formulation, is reported in Appendix 1.

Other experiments indicated that the oxide particles in chromium plate, which are visible under the microscope, remain essentially unchanged as to position during annealing treatments. There is a tendency toward breakup of the oxide films into spheroidized particles. However, no evidence was found that these particles migrate to the grain boundaries during annealing. Therefore, it is concluded that these visible oxides play little or no part in the annealing process. Photomicrographs illustrating these remarks are shown in Appendix II.

With this new information available, the possible factors causing hardness and ductility characteristics of chromium [illegible] were reinspected.

Grain Size

The extremely fine-grained nature of "as plated" chromium and the recrystallized structure of plates, softened by annealing, are believed to constitute conclusive evidence that the hardness of the plates is directly related to the grain size. The softening on annealing can be followed (through metallographic examination of structures) as a definite correlation between extent of recrystallization and resulting grain size and the hardness.

Internal Stress

Internal stress in chromium plate has not been studied so thoroughly as other factors. It is known that "as plated" chromium contains high internal stress which is relieved during annealing. The slight softening on heating hard plate to 300°C. may be explained as a result of relief of internal stress. X-ray photograms do not reveal this change in stress condition, since the fine-grained character of the plate produces diffuse lines before and after stress relief. It is certain that stress relief precedes recrystallization and, therefore, the temperature range for stress relief is fairly well established as being around 300°C.

Occluded Hydrogen

Previous work(1) has shown that the removal of occluded hydrogen does not appear to correlate in any way with the softening of chromium plate on heating. No additional work was done during the present contract period which would amplify the previous information.

Dispersed Oxide

The evidence now available indicates that the oxide in hard chromium plate is a result of the conditions causing hardness, rather than being itself the cause of the hardness. The lack of ductility in hard chromium plate, or in annealed chromium plate, is ascribed in the first case to the oxide films in the plate, and in the latter case to the spheroidized oxide particles which are lined up in the positions formerly occupied by the hydrated oxide films from which they are derived.

STATUS OF WORK ON PHYSICAL CHARACTERISTICS OF CHROMIUM PLATE

The information relating to the physical properties of chromium plate produced by presently known methods is believed to be sufficiently well developed to allow intelligent engineering application of the plate. Such applications would relate to low loads and moderate temperatures.

These factors show why conventional chromium plate cannot be expected to give the desired improvement in the tube life for hypervelocity firing and for large bore cannon. It is clear that the character of the chromium plate must be changed. Subsequent plating studies were undertaken toward improvements in the ductility of chromium plate.

STUDY OF CATHODE FILM CONSTITUENTS AND THEIR EFFECT ON FILM pH

A considerable amount of work directed toward the study of cathode film constituents was reported previously(1). In that work the cathode film material studied was prepared synthetically. In the present studies, attempts were made to obtain cathode film samples during actual plating operations. A hollow and porous nickel cathode was used and material was drawn from the hollow portion of the cathode while the outside was being chromium plated. By this method it was possible to obtain samples of solution drawn through the plating zone. The samples contained as much as 20 per cent of the chromium concentration in the partially reduced state (Cr^{+3}), whereas the bath contained a small fraction of 1 per cent of the contained chromium as Cr^{+3}. The pH of the samples was approximately 0.4 pH unit higher than that of the bath proper.

These experiments did not furnish the type of final information desired, but did provide evidence in confirmation of the cathode film theory previously advanced(1). The work was discontinued because there was no better sampling method available. The results of other experiments carried on during the project have indicated that no further work along this line was justified during this program.

SURVEY OF THE POSSIBILITIES FOR IMPROVING THE HOT-HARDNESS OF CHROMIUM PLATE

Hot hardness of chromium plate might be achieved by alloying, or by a conversion treatment such as nitriding or boronizing. Alloy plating experiments are described in a later section. Boronizing and nitriding treatments on chromium plate were carried out under supposedly optimum conditions. In both cases, the results were negative, indicating a diffusion rate too slow for practical operation within allowable temperature ranges for gun tubes.

Boronizing

The boronizing method consisted of passing hydrogen gas containing boron trichloride vapor over heated, chromium-plated specimens. Boron was deposited on the chromium surfaces and the specimens were then held at elevated temperature for short-time diffusion treatments. Details of time and temperature for plating and diffusion are given in Table 1. Further details regarding the plating method are available in the literature(2).

Metallographic examination of the boron plated specimens revealed no evidence of interdiffusion between the boron and chromium. It was concluded that the diffusion treatments were not sufficiently long to allow a significant amount of penetration. No prolonged diffusion treatments were undertaken because of the deleterious effect a prolonged high-temperature treatment would have on the gun tube material.

Nitriding

Initial nitriding tests were carried out in a muffle furnace, chromium plated specimens receiving the treatment at the same time a group of steel specimens were being nitrided. The nitriding temperature was 510°C.; the time 48 hours; and the degree of ammonia dissociation was maintained at 30 per cent. These specimens were examined under the metallograph and hardness measurements were made with the Tukon tester. There was no evidence of a nitrided layer, though the steel samples treated at the same time had satisfactorily hardened surfaces.

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(2) Parravano, N., and Mazzetti, C., Rec. trav. chim. 42, 821 (1923); C.A. 17, 3651 (1923).

New nitriding experiments were then conducted in a tube furnace, the temperatures being raised to 900°C. The treatment time was 24 hours. Metallographic examination revealed a layer at the surface which was adjudged to be chromium nitride.

Two layers were found at the interface between chromium plate and the steel basis. The layer in immediate contact with the steel appeared to consist of an iron-chromium diffusion alloy as shown by previous work. The layer between this and the chromium appeared to be chromium nitride.

The chromium nitride phase, Cr_2N, had been previously identified by X-ray tests on chromium plate samples heated in air for prolonged periods at 1100°C. Figure 4 presents X-ray diffraction data for those tests. The photomicrographs in Appendix II show the structure of the nitride layers formed. The similarity in appearance and structure of the layers found in the nitriding experiments and those in the air-heating experiments left no doubt as to their similarity in composition. Therefore, no X-ray tests were made on the nitrided samples.

These tests show that the nitriding of chromium plate occurs only at high temperatures, 1000°C. or higher being required to attain significant rates. Ordinary gun steels cannot tolerate long treatments at this temperature. However, the nitride coating is hard, and with a ductile backing might be of use in applications other than gun tubes.

It was shown that the nitride layer ends in a sharp interface at the unaffected chromium. This does not allow for a gradual change from the hardness of the nitride to the hoped-for ductility of underlying chromium. Therefore, no further work along these lines appears to be justified.

A SEARCH FOR AN IMPROVED CHROMIUM PLATING PROCESS

The tests described in the preceding sections and in reports of earlier work(1) have clearly shown that ductility and hot hardness may not be expected from conventional chromium plate. The lack of ductility is ascribed to the oxide films distributed through the plate, and, in turn, these films are related to the deposition and decomposition of chromium hydride during the plating operation. Therefore, means were sought to obtain a chromium plate which is deposited as body-centered, cubic chromium metal and not as a chromium hydride. Fused-salt baths or nonaqueous organic baths contain no hydrogen ion as such, and, therefore, baths of these types were studied with the hope of developing an improved plating process.

The experimental work reported in Appendix III is believed to cover the most obvious possibilities for improving the chromium plating process. The alloy plating field was largely avoided because of the time limitations of the contract period. The time available was mainly devoted to study of pure chromium plating baths with the idea of finishing this work during the existing contract period. The alloy plating work was set aside for later consideration in case the earlier efforts were not successful.

All of the efforts to improve the chromium plating process were unsuccessful. Thick deposits from fused salt baths were invariably powdery or granular. No way has been devised to obtain buildup of coherent ductile chromium metal.

STATUS OF WORK

The new information cited in the preceding sections shows that as-deposited chromium from the chromic acid bath cannot be expected to have either ductility or hot hardness. Also, extensive studies of alternate types of baths have not developed a means of producing a ductile plate nor have nitriding and boronizing tests indicated a practical method for inducing hot hardness in chromium plates.

These results show cause for the poor performance of chromium plate in extreme conditions of gun tube service but do not offer means for improving the performance. It is now clear that the desired improved ductility and hot hardness must be sought in chromium rich alloys. A number of such alloys show promise, and the high chromium-iron alloys are particularly interesting. Favorable results have been reported for studies with breech liners of forged chromium-iron alloy. Electrodeposition of such alloys would allow protection of the entire gun tube. Recent work(3) indicates strong possibilities for successful deposition of high chromium-iron alloys. Combinations of chromium and the various refractory metals are other alloys which appear to be worthy of study.

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(3) Pilot-Plant Production of Electrolytic Chromium, Lloyd, Rosenbaum, Homme, and Davis, Jour. Electrochem. Soc. 94, 122 (1948).

APPENDIX I

This section of the report presents Figures 6, 7, and 8, which show additional data on recrystallization of conventional hard chromium plate at elevated temperatures.

APPENDIX II

This section of the report presents Figures 9 and 10, which show the change in "oxide inclusion" when conventional hard chromium plate is heated.

APPENDIX III

This section of the report contains the essential data and results of plating experiments to explore the possibilities for an improved chromium plating process.

APPENDIX III

Apparatus

Electrolysis of fused salt baths and organic chemical baths was carried out in vertical muffle electrically heated furnaces. Temperatures were controlled by recorder controllers in some cases and by manual adjustment of variable transformers in other cases. Temperatures of low-temperature baths were read on thermometers immersed in the baths. The temperatures of high-temperature baths were read from recorder-controller charts. Most of the baths were contained in glass beakers except for a few cases where refractory crucibles were required.

Aqueous baths were electrolyzed in glass beakers with electrical immersion heaters controlled by thermoregulators.

In some experiments, alternating current was superimposed on the direct plating current. The circuit used for this purpose is illustrated in Figure 5. In other experiments, a high-frequency magnetostrictive vibrator was used to mechanically vibrate cathodes. The frequency was 9000 cycles per second, and the amplitude was about 0.001 inch. For low-frequency vibration, a Traynor magnetic vibrator was used. This type of equipment is used commercially as a feeding device for granular materials.

For most of the following work, sheet metal cathodes one inch square were used. When cathodes were spun or vibrated, wire or rod material was used.

Experimental Procedures

Table 2 outlines the experimental data obtained. The following notes, titled according to bath type, extend the information available in the table.

A. Fused Salt Baths.

1. Chloride Baths. Of the ten bath compositions tried in this classification, a coherent deposit was obtained from only one (Bath No. 8 in Table 2). The other baths yielded powders or no deposit at all. The plates from Bath No. 8, while coherent, were so poor in physical properties that no further work with the bath was considered. It is likely that the deposits contained much aluminum.

2. Cyanide Baths. The three baths investigated under this classification (Baths No. 11, 12, and 13) gave either no deposit or only a trace of chromium powder. At high current densities, metallic sodium was liberated and rose to the bath surface.

3. Thiocyanate Bath. The only bath tried in this category (No. 14) yielded only a light powder deposit on the cathode in 30 minutes of electrolysis. The bath was tried because its operating temperature is low compared to the cyanide baths.

4. Fused Baths of Mixed Salts. In this classification are chloride-type baths used in an attempt to plate chromium-tungsten alloy, and a mixed cyanide-chloride bath for deposition of chromium alone. None of these baths yielded a promising deposit. Powder deposits were produced in all cases, sometimes underlaid with a coherent flash of chromium.

B. Organic Type Baths.

1. Urea Baths. Baths formulated with urea, ammonium chloride, and chromic chloride were reported to have produced chromium plates in an English laboratory. The baths are fused and operated at temperatures from 300 to 400°F.

In the present work, flash plates with a metallic appearance were obtained from these electrolytes. Prolonged electrolysis did not significantly increase the thickness of the plates, which were very thin in most cases, and, when thickness was obtained, an organic compound appeared to be deposited with, or instead of, the metal. A carnauba wax layer was placed on the surface of Bath No. 21 in the hope of excluding air. However, the deposits were not benefited.

An alloy plating attempt was made (Bath No. 28) by adding ferrous bromide to the urea type bath. Again an organic compound was deposited, but this time some free iron was found in the deposit. In a few experiments, some chromium was also found in the deposits, but in all cases the amount of chromium was under one per cent. One experiment yielded a deposit which was 91.5 per cent iron.

2. Formamide Baths. The use of formamide type baths to obtain chromium plates is reported in the literature(4). The baths are saturated with HCl by bubbling anhydrous HCl through them before the electrolysis is begun.

In the present work, the HCl gas was bubbled through the baths for 2 hours before electrolysis and the gas flow was continued during electrolysis. As indicated in Table 2 (Bath No. 29), no satisfactory chromium deposits were obtained. Attempts to build up the plate always resulted in a powdery deposit of metal.

3. Quaternary Ammonium Compound Baths. Of the two quaternary compounds tried in experimental chromium plating baths, one produced strictly flash plates (Baths No. 30 to 33) and the other, black non-conducting cathode films or no plate of any kind (Bath No. 34). The two compounds were, respectively, tetraethanol ammonium hydroxide and ethyl pyridinium bromide.

An effort was made to formulate a bath of chromic chloride-ethyl pyridinium bromide in the same molar concentrations as those used by a

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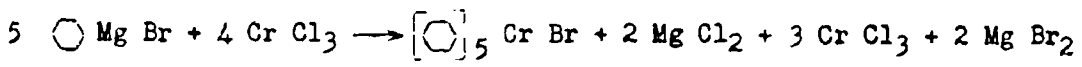
(4) Blue and Mathers, Trans. Electrochem Soc. 63, 231 (1933).

previous investigator(5) to deposit aluminum from a bath of aluminum chloride-ethyl pyridinium bromide. Unlike the aluminum bath, this one at two moles of chromic chloride per mole of ethyl pyridinium bromide was found to be solid up to decomposition temperatures. When lower concentrations of chromic chloride were used in order to obtain a melt, the bath showed poor conductivity and gave black deposits.

Flash plates were obtained when a bath containing 10 grams of anhydrous chromic chloride in 100 grams of tetraethanol ammonium hydroxide was electrolyzed.

4. Grignard Reagent Baths. For the preparation of the one bath in this classification, an excess of magnesium was reacted with bromo benzene in anhydrous ether. The product in ether solution was removed from the magnesium and reacted with the approximate stoichiometric weight of chromic chloride for the reaction:



The resulting mixture had high electrical resistivity and tended to solidify. No plate resulted when electrolysis was attempted.

Though the result was unfavorable in this experiment, experimentation on Grignard reagents for chromium and chromium alloy plating should be investigated further.

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(5) Weir, The Electrodeposition of Metals in Fused Quaternary Ammonium Salts, Master's Thesis, Rice Institute (1942).

5. Other Organic Baths. A flash plate was obtained from a melt of tris (hydroxymethyl) aminomethane with anhydrous chromic chloride. From baths with anhydrous chromic chloride and thiourea dark, nonconducting cathode films resulted.

C. Aqueous Baths.

1. Chromic Acid Baths. Experiments with conventional chromic acid baths were conducted to test two variations from standard plating conditions; extreme vibration, and reduced pressure. The rod cathodes were vibrated at 9000 cycles per second with a magnetostrictive type high-frequency apparatus, and at 60 cycles per second with a magnetic vibrator. The deposits appeared to be affected by the high-frequency vibration, in that oxide films were shorter than normal in the microstructure. However, the deposits tended to spall from the basis metal, so the work was discontinued. The 60-cycle vibration had little or no effect on the structure of the plates.

Plating at reduced pressure was suggested by a recently reported research(6), which reported that the efficiency of cyanide copper plating was greatly improved by reduced pressure on the bath surface. One experiment with a standard chromic acid bath at a temperature of 85°F. and a

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(6) The Effect of Pressure on Current Efficiency of Copper Electrodeposition from Cyanide Solutions, paper to be presented before October, 1948, meeting of Electrochemical Society and subsequently published by that Society.

pressure of approximately 10 mm. of Hg gave a cathode efficiency of approximately 22 per cent. Attempts to plate at more elevated temperatures required apparatus changes which were not yet successfully completed at the close of the contract period. It is not possible to say that the preliminary indications of increased efficiency are correct until more data are available.

2. Fluoborate Baths. An alloy of chromium-iron was deposited from a fluoborate bath (Baths No. 48 to 54) which was made up with one volume each of chromic fluoborate and ferrous fluoborate concentrates (assay approximately 45 per cent metal fluoborate) along with two volumes of water. The most favorable operating cathode current density was 288 amperes per square foot. The temperature of the bath varied from 85 to 120°F. The deposit was improved by the addition of 0.20 cc. quinoline to 100 cc. of bath, a plate of lighter color being produced.

When the plate was examined by X-ray diffraction methods, a diffuse iron pattern was obtained. Chemical analysis showed that the plate contained 3 to 5 per cent chromium. Aside from the chromium content being less than desired, the plate was spongy and could easily be scraped from the cathode. The replacement of quinoline with glue (Baths No. 55 and 56) yielded a solid, homogeneous plate which was very brittle.

3. Other Aqueous Baths. None of the three baths listed showed any promise.

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October 22, 1948

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INDEX OF FIGURES AND TABLES

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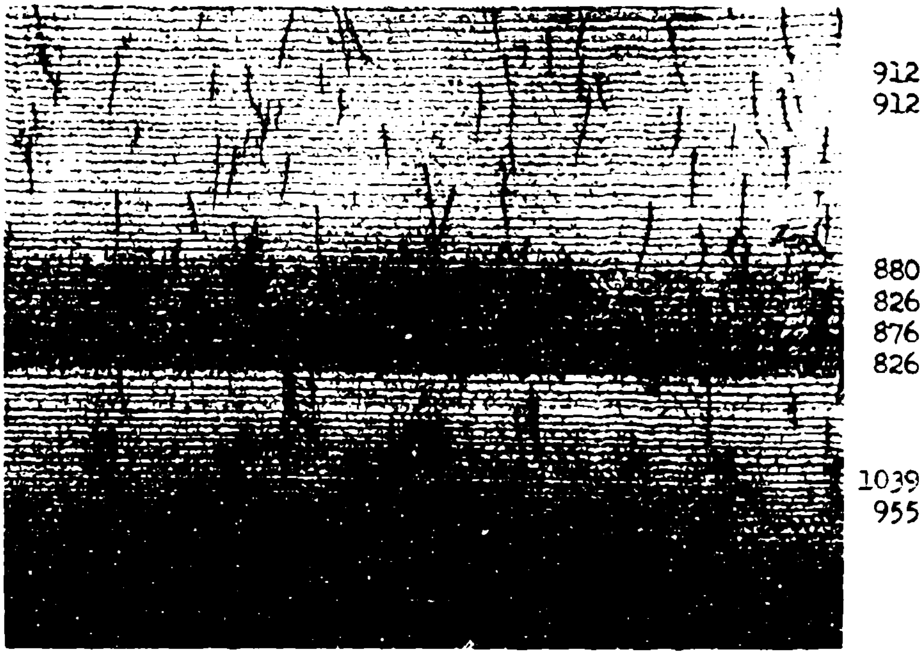

HCl etch

54493
300X

Figure 1. Cross-sectional view of hard chromium plate in the "as-plated" condition. Vertical lines are inclusion-filled cracks. Horizontal lines are unexplaired striations within the plate. Figures at right-hand margin are Knoop hardness numbers for the locations shown.

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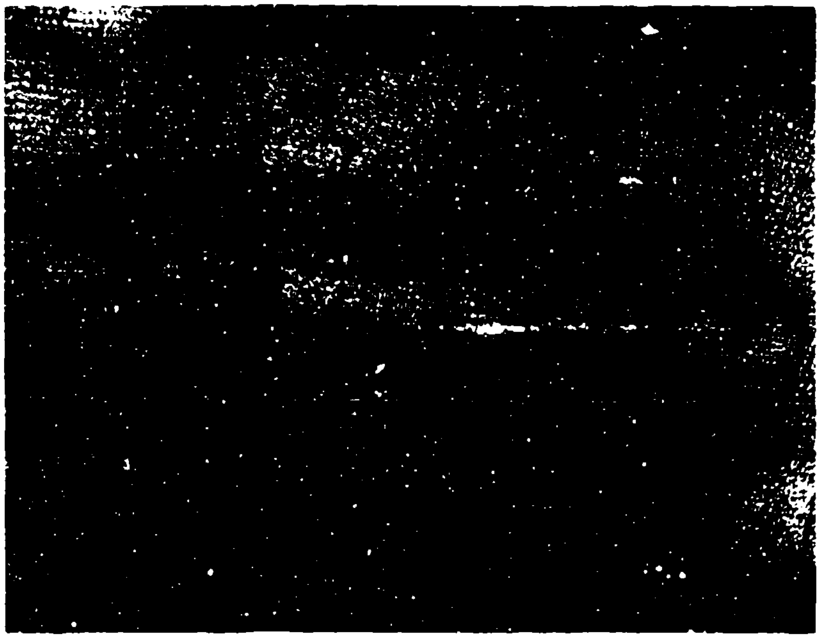
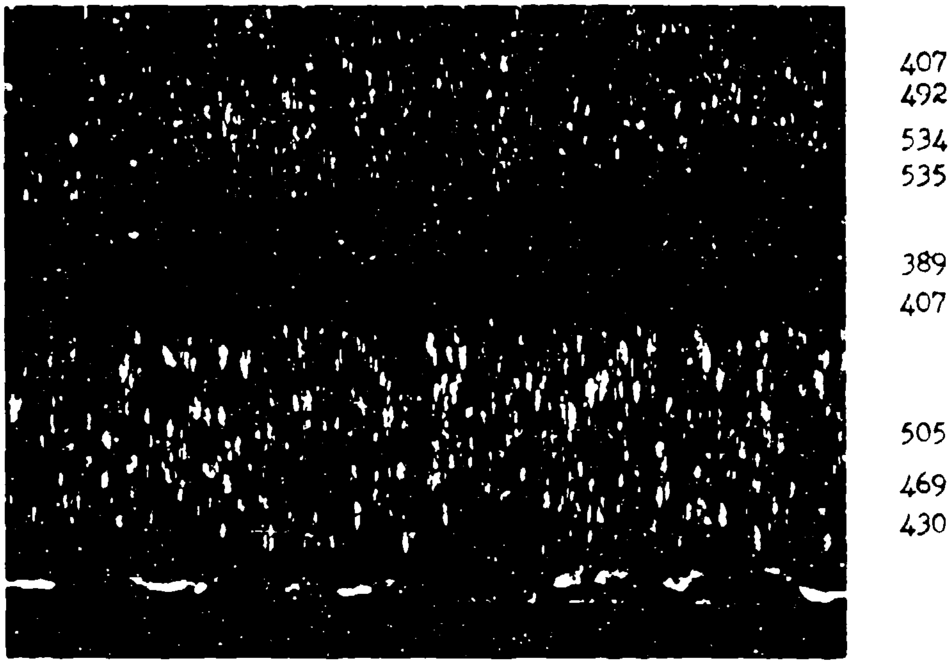


HCl etch

54495
300X

Figure 2. Cross-sectional view of hard chromium plate after 22-hour treatment at 300°C. Striations are still visible though the etch did not bring them out so strongly as in the "as-plated" sample of Figure 1. No evidence of re-crystallized grains is visible. Figures at right-hand margin are Knoop hardness numbers for the locations shown.



HCl etch

54496
300X

Figure 3. Cross-sectional view of hard chromium plate after 12-hour treatment at 500°C. Recrystallized grains are plainly visible as the white elongated areas set in the darker background. Note that the striations are still faintly visible and that some of the recrystallized grains reach through several layers of striations. Figures at right-hand margin are Knoop hardness numbers for the locations shown.

TABLE 1. DATA FOR BORON PLATING TESTS

Sample No.	Plating T, °C.	Plating Time (Min.)	Diffusion T, °C.	Diffusion Time (Min.)
3561-4A	800	5	800	20
3561-4B	1000	5	1000	5
3561-4C	1200	5	-	-

Note: Boron deposited by bubbling H_2 through BCl_3 at -22°C., then directing it over the heated specimen to be plated.

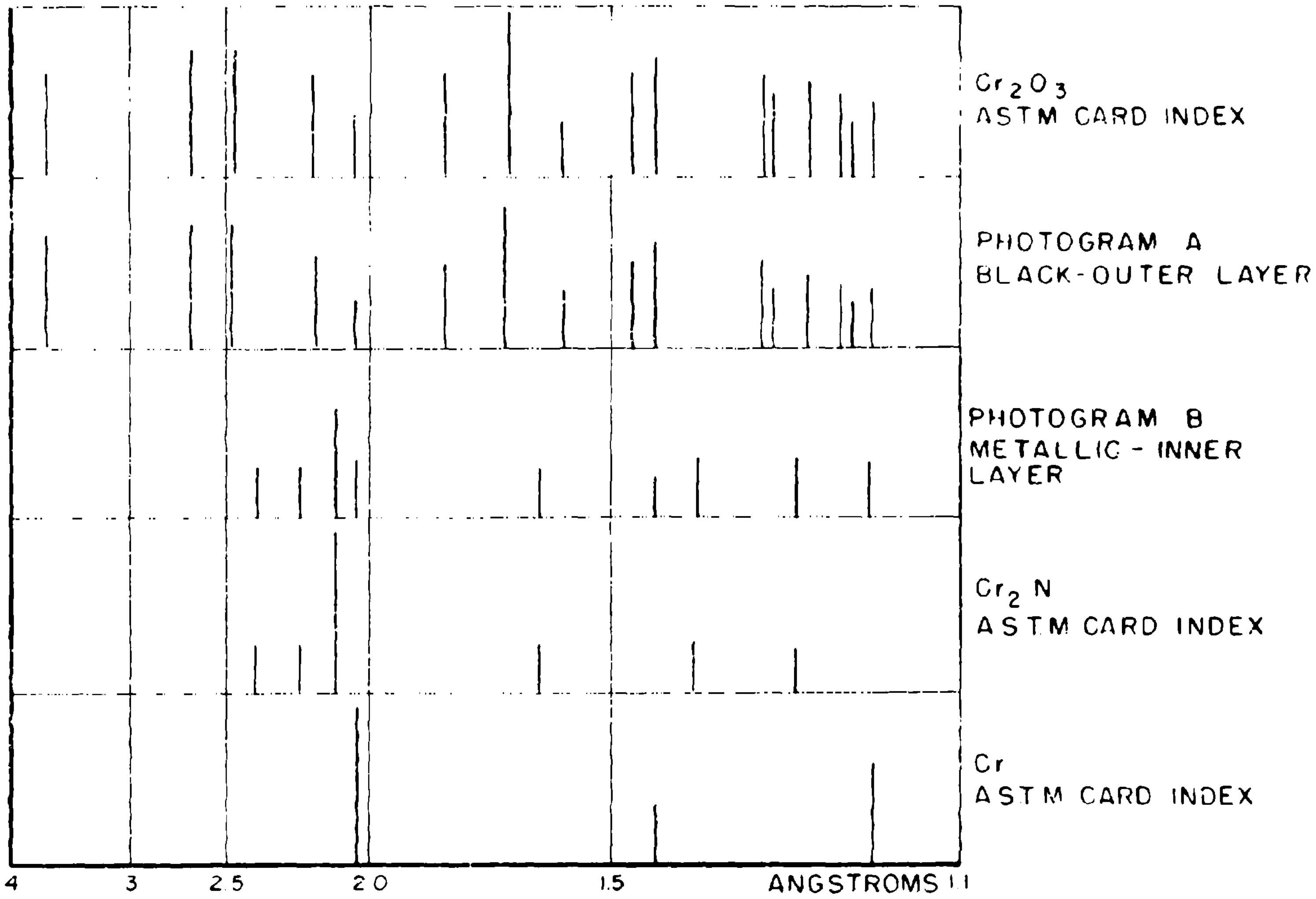


FIGURE 4. GRAPHICAL REPRESENTATION OF X-RAY DIFFRACTION DATA. THE ABSCISSAE REPRESENT INTERPLANER SPACINGS IN ANGSTROM UNITS. THE ORDINATES REPRESENT THE RELATIVE INTENSITIES.

0-9585

NOTE METERS SHOWN WERE KEPT IN THE CIRCUIT ONLY D.C. OR A.C. RESPECTIVELY WAS IMPRESSED ON CIRCUIT WHEN METERS WERE READ. READINGS SERVED ONLY TO STANDARDIZE CONDITIONS.

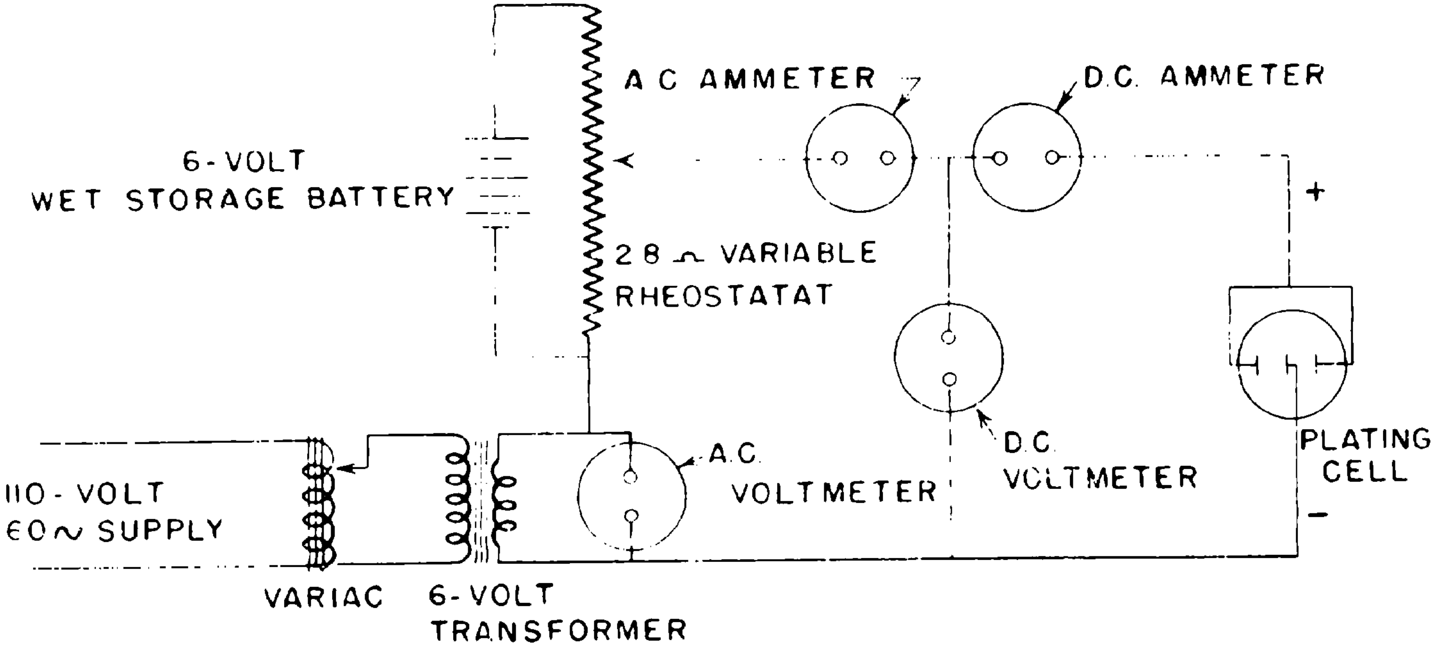
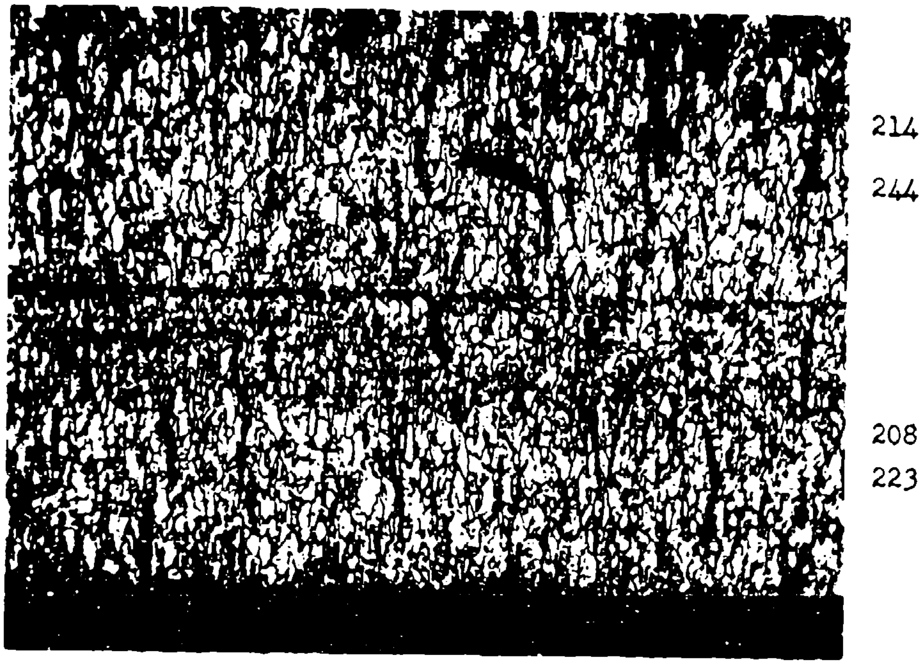


FIGURE 5. CIRCUIT FOR SUPERIMPOSING 60-CYCLE A.C. ON D.C.

O-9586



HCl etch

54497
300X

Figure 6. Cross-sectional view of hard chromium plate after 12-hour treatment at 700°C. The heat treatment has completely recrystallized the plate. Figures in right-hand margin are Knoop hardness numbers for locations shown.

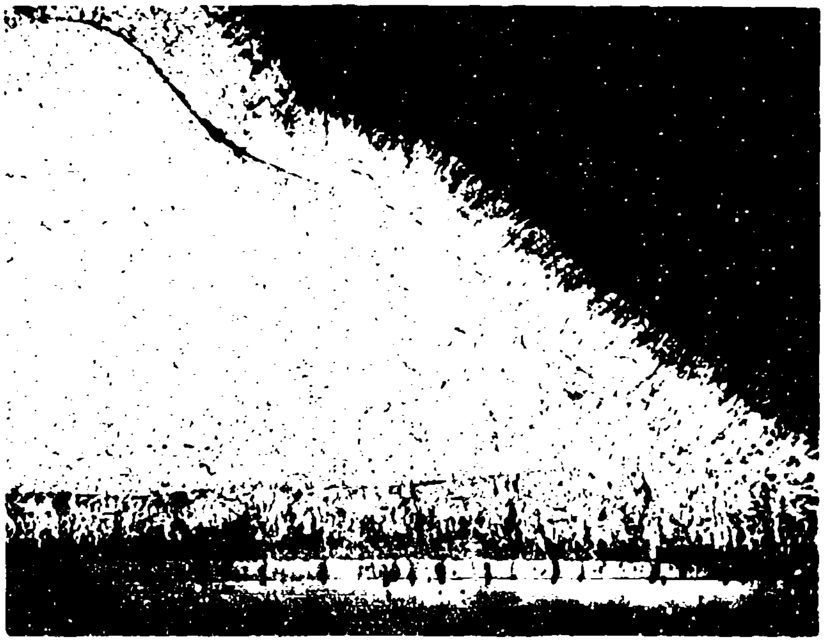
HCl etch

54498
300X

Figure 7. Cross-sectional view of hard chromium plate after 12-hour treatment at 900°C. Little or no change from the structure of Figure 6 (700°C. annealing) has occurred. Note that an alloy layer has formed between the steel basis metal and the chromium plate. Figures in right-hand margin are Knoop hardness numbers for the locations shown.

HCl etch 54499 200X

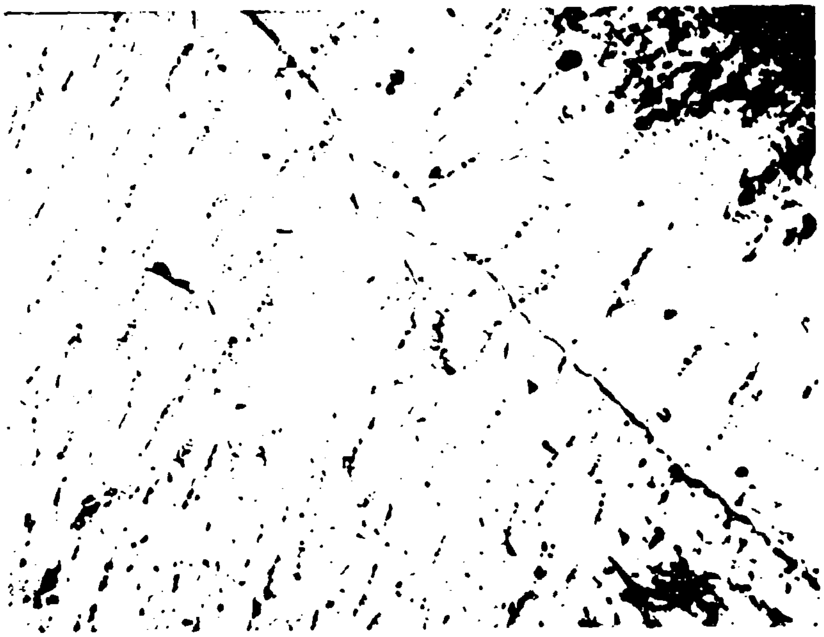
Figure 8. Hard chromium annealed at 1100°C. for 12 hours. Grains have now become nearly equi-axed. Inclusions are clearly shown to be in original positions with no evidence of migration into grain boundaries. Note the nitride penetration along the large vertical crack in the plate.



As polished

54502
75X

Figure 9. Cross-sectional view of hard chromium plate annealed at 1100°C. for 60 hours. The separate layers shown are, from the bottom up; steel basis metal, iron-chromium diffusion layer, chromium nitride layer, recrystallized chromium plate, and chromium nitride. The chromium nitride layer surrounds the recrystallized chromium plate on all sides. Figures in right-hand margin are Knoop hardness numbers for successive layers.



As polished

54503
250X

Figure 10. Same plate shown in Figure 9. View shown is at boundary between recrystallized chromium plate (lower left) and chromium nitride (upper right). Note that major portion of oxide particles are globules strung out in lines, indicating that they are probably dehydrated inclusion films.

TABLE 2. SUMMARY OF PLATING EXPERIMENTS

	Bath						
Bath No.	Material	Amt. (Gms.)	Material	Amt. (Gms.)	Cr Source	Amt. (Gms.)	Temp. (°F.)
			FUSED SALT BATHS Chloride Baths				
1.	KCl	137	LiCl	113	$CrCl_3$	3	900 950 "
2.	"	"	"	"	$Cr(BF_4)_3+$ $CrCl_3$	- 3 "	900 "
3.	KCl	137	LiCl	113	$CrCl_3$	2.5 "	1000 800
4.	"	47	"	65	$CrCl_2$	15	900 850 800
5.	NaCl	200	LiCl	200	$CrCl_3$	10	1150 1260 " "
6.	NaOH	280	NaCl	88	Cr_2O_3	228	1000
7.	$AlCl_3$	234	NaCl+ KCl	31 28	$CrCl_3$	3	320 280 200 " 250
8.	"	200	NaCl	85	"	10	530 " 600 430 400

Anode Material	Deposit		Remarks
	Amount	Type	
Graphite	Some	Mostly powder	3 Volts AC superimposed
"	"	Dense powder	" " " "
"	"	Powder	" " " "
"	Heavy	"	Spinning rod cathode
"	Some	"	" " "
"	Heavy	Powder	Slowly turning rod cathode
"	Some	"	" " " "
"	Small	Powder	
"	"	Coarse Powder	
"	"	Coherent Powder	Spinning rod cathode
Hard Carbon	None	---	Temperature too low
Ditto	"	---	
"	"	---	
"	"	---	
"	None	---	Bath mushy
"	"	---	Cathode corroded; 3 volts AC superimposed on DC
"	"	---	"
"	"	---	"
"	"	---	3 Volts AC superimposed on DC
"	Some	Powder	" " " " " "
Hard Carbon	"	Coherent Plate	
Ditto	.001 inch	" "	
"	"	" "	
"	Flash	Flash	
"	Some	Coherent Plate	
"	Flash	Flash	3 Volts AC superimposed on DC

Anode Material	Deposit Amount	 Type	Remarks
Graphite	Some	Mostly powder	3 Volts AC superimposed
"	"	Dense powder	" " " "
"	"	Powder	" " " "
"	Heavy	"	Spinning rod cathode
"	Some	"	" " "
"	Heavy	Powder	Slowly turning rod cathode
"	Some	"	" " " "
"	Small	Powder	
"	"	Coarse Powder	
"	"	Coherent Powder	Spinning rod cathode
Hard Carbon	None	---	Temperature too low
Ditto	"	---	
"	"	---	
"	"	---	
"	None	---	Bath mushy
"	"	---	Cathode corroded; 3 volts AC superimpos on DC
"	"	---	"
"	"	---	"
"	"	---	3 Volts AC superimposed on DC
"	Some	Powder	" " " " " "
Hard Carbon	"	Coherent Plate	
Ditto	.001 inch	" "	
"	"	" "	
"	Flash	Flash	
"	Some	Coherent Plate	
"	Flash	Flash	3 Volts AC superimposed on DC

TABLE 2. (CONTINUED)

	Bath						
Bath No.	Material	Amt. (Gms.)	Material	Amt. (Gms.)	Cr Source	Amt. (Gms.)	Temp (°F.
FUSED SALT BATHS (CONTINUED) Chloride Baths (Continued)							
9.	$AlCl_3$	280	NaCl	120	$CrCl_3$	10	500
							"
							"
							450
							"
10.	$AlCl_3$	280	NaCl	120	$CrCl_3$	10	500
							"
							"
Cyanide Baths							
11.	NaCN	172	KCN	98	Cr_2O_3	32.2	950
12.	"	"	"	100	"	12	1100
							"
13.	NaCN	150	KCN	130	$CrCl_3$	10	1000
Thiocyanate Baths							
14.	KCNS	100	KOH	100	Cr_2O_3	100	550
Mixed Salt Baths							
15.	$AlCl_3$	280	NaCl $Na_2WO_4 \cdot 2H_2O$	120 100	$CrCl_3$	50	500
16.	"	"	NaCl $Na_2WO_4 \cdot 2H_2O$	120 40	$CrCl_3$	25	"
17.	"	"	"	"	"	"	"

Amt (Gms.)	Temp. (°F.)	Amp./Ft.^{2}	Time (Min.)	Voltage	Cathode Metal	Anode Material	Deposi
							Amount
10	500	4.3	30	1.7	Copper	Hard Carbon	None
	"	6.1	"	1.9	"	Ditto	Flash
	"	5.4	"	2.1	"	"	"
	450	4.6	120	2.2	"	"	"
	"	2.9		2.2	"	"	"
10	500	14	120	1.5	Copper	Chromium	Heavy
	"	7	30	1.2	"	"	Some
	"	5	"	"	"	"	"
32.2	550	186	120	3	Iron	Graphite	None
12	1100	"	"	"	"	"	"
	"	"	720	"	Nickel	"	"
10	1000	64	60	1.7	Copper	Chromium	Some
100	550	186	30	3	Nickel	Graphite	Small
50	500	36	30	-	Copper	Tungsten	Some
25	"	21	720	2.1	"	"	Heavy
"	"	36	60	2.3	"	"	"

Anode Material	Deposit		Remarks
	Amount	Type	
Hard Carbon	None	---	$CrCl_3$ settled out
Ditto	Flash	Flash	" " "
"	"		Some powder deposited
"	"	"	" " "
"	"	"	" " "
Chromium	Heavy	Powder	Some insoluble $CrCl_3$, Cathode Vibrated at 60 cycles per second
"	Some	"	" " " " " "
"	"	"	" " " " " "
Graphite	None	---	Iron cathode corroded in bath Some sodium was liberated
"	"	---	Cathode corroded away
"	"	---	" " "
Chromium	Some	Powder	Vigorous reaction on mixing 3 Volts AC superimposed
Graphite	Small	Powder	Cathode corroded away at bath surface in a longer run
Tungsten	Some	Powder	Cathode vibrated at 60 cycles per second
"	Heavy	Powder Slight Flash	" " " " " 3 Volts AC superimposed
"	"	" "	" " " "

END

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