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Prevention of Significant Embrittlement in Certain Types of High-Strength Steels, Prior to and During Cadmium Electroplating[1]

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In the past, high-strength steels have become seriously embrittled during surface pretreatment and during subsequent cadmium electroplating. During this research, a method of chemical pretreatment was discovered by means of which the surfaces of Type 1062 and 4037 steels may be smoothed to eliminate sharp edges and cleaned for plating without introducing a significant amount of embrittlement. It also was found that specimens pretreated in this manner and plated with cadmium by a procedure previously developed by the authors are essentially free from embrittlement. Considerable success also has been obtained with Type 4340 steel. A chemical method of removing embrittlement and plating without introducing further embrittlement also was discovered. Evidence is presented to suggest that this type of embrittlement is primarily a surface phenomenon.

Ordinary steels frequently have been protected from corrosion by the use of electroplated cadmium coatings. However, serious embrittlement problems have been experienced when attempts have been made to cadmium plate high-strength steels for the same purpose.

In 1961, Geyer, Lawless, and Cohen (1) stated, "Hydrogen embrittlement as a potential source of failure of steel has long been recognized as a serious problem. . . . Electroplating processes are recognized as the most common source of detrimental hydrogen embrittlement and cadmium electroplating processes are considered to be among the most serious offenders. . . . The Air Force presently does not permit cadmium electroplating of certain steels having strengths exceeding 220,000 psi. It has been necessary, therefore, for manufacturers to resort to other techniques, inferior to cadmium plating, for applying corrosion preventive coatings. . . . The numerous methods used for studying hydrogen embrittlement, coupled with the various methods used for testing for hydrogen embrittlement effects, have caused much confusion and frustration in the metal-finishing industry." This situation has not changed greatly since 1961. It is true that embrittlement often can be reduced by baking; however, this additional step is time consuming, adds to the cost of the finished article, and frequently is not completely effective. While baked experimental specimens were produced and tested during the work described by Geyer, Lawless, and Cohen, it should be emphasized that the specimens used during the research reported in the present paper were not baked.

The seriousness of the embrittlement that may occur when high-strength steels are pretreated and electroplated was demonstrated by experiments conducted in this laboratory. In each one, 12 pins of AISI Type 1062 high-carbon steel were given either a preparatory treatment alone or a preparatory treatment + cadmium plating in a standard commercial bath having the composition given in Table I. After the completion of these various treatments, each of the pins was bent in the Hounsfield testing machine, described later in this paper, to determine the extent of the embrittlement that had taken place. Highly embrittled pins could not be bent through as large an angle as slightly embrittled ones without breaking. The results of these experiments are compared in Table II with those obtained when untreated pins were bent.

[1] Submitted for publication August 5, 1965. Crown Copyright Reserved.

Table I. Compositions of cadmium plating baths (g/liter)

Constituent	Commercial bath (unstable)	Stable bath prepared according to formula of Dingley and Bednar
NaCN	124.5	98.3
NaOH	9.6	80.3
Cd	17.1	34.5
Na_2CO_3	25.5	<6.0
Ratio $\frac{NaCN}{Cd}$	7.3	2.8
Rohco 100 brightener	0.8	0.8
Rohco X1 20 brightener	0.2	0.2

John Bednar studied at the Free Ukrainian and Munich Universities in Munich, Germany, and also at Carleton and Ottawa Universities in Ottawa, Canada. He has done research for five years at the Canadian Dept. of Mines and Technical Surveys to improve the methods of electroplating zinc, cadmium, and copper from cyanide baths. He is author or coauthor of a number of published papers.

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Raymond R. Rogers received his M.A. degree in electrothermics at Toronto University in 1926, and his Ph.D. in electrochemistry at Columbia University in 1933. He continued there as instructor and research supervisor until 1941. Since 1944, he has been in charge of research in corrosion and its prevention and in pyrometallurgy at the Canadian Dept. of Mines and Technical Surveys.

Table II. Results of embrittlement experiments

Expt. No.	Nature of treatment	Percentage of pins broken at angles less than 135°	Range of angles at which breaking occurred (°)
1	None	17	85-86
2	Sulfuric acid	100	8-35
3	HCl	50	65-120
4	Hydrochloric acid + cathode treatment in sodium hydroxide solution	33	55-101
5	No. 4 + cadmium plating	100	11-32

In the present research, an effort was made to find a method of pretreating and cadmium electroplating that would not appreciably embrittle high-strength steels of AISI Types 1062, 4037, and 4340. Type 4037 high-strength steel is more sensitive to embrittlement than Type 1062, and Type 4340 is even more sensitive.

Dingley and Bednar (2) had reported the discovery of a method of preventing the serious embrittlement of AISI Type 1062 high-strength steel wire of Rockwell C hardness 52-56 during acid treatment and zinc electroplating. They had found that the following precautions are required to insure freedom from this embrittlement:

1. The surface of the steel must be adequately prepared before the zinc is deposited,
2. The plating must be done in a properly formulated bath.

They recommended the use of hydrochloric acid pickling accompanied by ultrasonic vibration prior to plating, and the use of a stable zinc cyanide plating bath, *i.e.* one in which OH normality = 1.2 + total CN normality. No afterplating baking treatment was required when these precautions were followed and when an appropriate cathode current density was maintained during the plating. Dingley and Bednar (3) also had shown that a cadmium plating bath could be kept in a stable condition by maintaining the relationship OH normality = total CN normality.

Since stable baths were known to be available, it was hoped that the present problem would be solved when a method had been found by which steel of Types 1062, 4037, and 4340 could be adequately prepared prior to plating. After this actually had been achieved in the case of Type 1062 and 4037 steels, a number of samples of these two steels were pretreated and immediately plated without significant embrittlement by the methods that had been developed. Last, it was shown that highly embrittled Type 1062 and 4037 steels may be readily treated to remove the significant embrittlement and then plated without further embrittlement. Although less success was achieved with the Type 4340 steel, it is felt that considerable progress was made.

The authors realize that much research must still be done in this field, and they consider this present paper as a progress report rather than a final one.

Experimental Materials and Equipment

The steels of Types 1062 and 4037 used in the present research had been supplied in the form of pins, and the degree to which each of these was embrittled during an experiment was expressed in terms of the angle of bend required to produce rupture. On the other hand, the Type 4340 steel had been supplied in the form of rods, and the degree of embrittlement of this material was expressed in terms of the length of time required to produce rupture in a specially prepared bar of the notch stretch rupture type. The bars were stretched by a load calculated on the basis of 75% of the ultimate tensile strength of the steel, the latter value having been determined in the machined and heat-treated condition prior to surface pretreatment and plating.

The pins of Type 1062 steel were 3.25 in. in length and 0.15 in. in diameter. They had been drawn from wire and austempered in the bainitic range, resulting in a Rockwell C hardness of 52-56 and an ultimate strength of 257-279 kpsi. The material was covered with a uniform thin scale of blue iron oxide which could be removed by treatment in dilute hydrochloric acid.

The pins of Type 4037 steel were 2.63 in. in length and 0.15 in. in diameter. They had been drawn from wire and then quenched and tempered, resulting in a Rockwell C hardness of 51-55. They were covered with a thin, very porous film of copper which had been applied to prevent decarburization during heat treatment, together with a certain amount of smutty, black material. During the first part of the program, these undesirable materials were completely removed by treatment in an aqueous solution containing 36% H_3PO_4, 23% HNO_3, and 24% CH_3COOH (by weight) before the pins were used experimentally. Later, it was found that these materials could be removed successfully by treatment in the HNO_3-CH_3COOH solutions that had been developed.

The test bars of Type 4340 (rough-machined) steel had been hardened in a neutral salt bath at 1500° ±25°F for 30 min and quenched in oil maintained at 75°-140°F. They then were tempered at 400° ± 10°F for 4 hr and air-cooled, resulting in a Rockwell C hardness of 50-54. After finish machining, the bars were stress-relieved at 375° ± 25°F for 4 hr. The 60° notch with a radius of 0.005 ± 0.001 in. then was machine-cut into the test bar, producing a diameter of 0.1768 ± 0.001 in. The ultimate strength of five test bars, chosen at random, ranged between 323 and 340 kpsi with an average of 332 kpsi. The metal at the notch was free from any visible film of oxide or other nonmetallic material.

The compositions of these three types of high-strength steels are given in Table III.

The equipment used in this investigation was as follows:

Sonogen ultrasonic generator Model LG-150, 25 kc, 150w, equipped with an LT-60 transducerized tank. Branson Ultrasonic Corp.

Daniels motor-driven portable plating barrel Model 35, 6 rpm. (Volume of plating solution used, 8 liters.)

Hounsfield notched bar bending jig attached to a tensometer machine Type K. During testing, the unnotched pin is supported at two points 1.19 in. apart. Pressure applied to a 0.125-in. diameter mandrel forces the pin into the gap between these two points until it breaks or until a bend of 90° has been produced at the end of 7 min. The jig used in this work had been modified to give a maximum bend of 135° in 11 min.

Satec creep and stress rupture machine, Special Model C, max capacity 12,000 lb.

Lorco Liquamatte wet-blasting equipment Model 22. Line pressure 80 lb/in.².

Experimental Procedures

Between 12 and 40 pins of Type 1062 or 4037 steel, or 1 or 2 bars of Type 4340 steel, were treated in each surface pretreatment experiment. These experiments were performed at room temperature in 600-ml glass beakers containing 300-400 ml of aqueous solution.

Table III. Analyses of steels (%)

Constituent	Type of steel (AISI) 1062	4037	E4340
C	0.64	0.40	0.38
Mn	1.03	0.76	0.78
P (max)	<0.02	<0.02	<0.02
S (max)	0.02	0.02	<0.01
Si	—	0.31	0.31
Ni	—	—	1.71
Cr	—	—	0.77
Mo	—	0.26	0.21

The concentrations (by weight) of the solutions used were as follows:

Sulfuric acid (H_2SO_4) — 16%
Hydrochloric acid (HCl) — 20%
Nitric acid (HNO_3) — 15%
Nitric acid (HNO_3) } A — { 15% ; B — { 12%
Acetic acid (CH_3COOH) } A — { 4% ; B — { 34%

Copper sulfate solution for copper immersion coating ($CuSO_4 \cdot 5H_2O$) — 10% (pH = 2.6)

City tap water was used in the preparation of these solutions as well as in all rinsing.

All cadmium electroplating experiments were performed in the plating barrel already referred to. Each charge in the barrel consisted of 12-40 pins, or 12-40 pins together with 1 or 2 tensile test bars. In every case, the plating was done either in a bath of the unstable commercial type or in one of the stable type recommended by Dingley and Bednar (3). The compositions of both of these baths are given in Table I.

In every plating experiment, the cadmium film was built up to 1 mil or more in thickness since experience has taught that no additional embrittlement would occur at thicknesses greater than that value. In some cases, the film was built up to about 8 mils so that the quality of the adherence of the cadmium to the steel could be determined.

When working with Type 1062 and 4037 steels, each surface pretreatment investigated was evaluated with regard to the cleanliness and smoothness of the pins produced using scales from "excellent" to "poor," and with regard to the degree of embrittlement of the pins as indicated by the angle of bend at which they were fractured. Each procedure involving plating was evaluated with regard to the last-mentioned characteristic only. When working with Type 4340 steel, the test bars were evaluated with regard to embrittlement only. The term "excellent cleanliness" was used in cases where the steel surface was very active as shown by the fact that rapid, uniform rusting appeared immediately after the surface was exposed to the atmosphere. When such a surface was placed in a plating bath, a uniform coating would form on it immediately after the plating circuit was closed. The term "excellent smoothness" was used in cases where the steel surface appeared to be free from the minute, sharp edges which can be seen on many steel surfaces after treatment when inspected at a magnification of 60X.

Experimental Results

The experiments referred to below are only a small proportion of the total number performed during the research. Only those were included that seemed to contribute to the clarity of the paper.

Types 1062 and 4037 Steels.—In the first series of experiments, the effects of surface pretreatments involving sulfuric acid with and without ultrasonics, and hydrochloric acid with and without ultrasonics, were investigated. The results of Expt. 6-11, presented in Table IV, show that the Type 1062 steel: (*a*) was embrittled during all of the procedures used except those involving hydrochloric acid with ultrasonics, the 1-min procedure of this latter type giving as good results as the 3-min one; (*b*) was not greatly improved in cleanliness by any of the procedures except those involving hydrochloric acid and even these did not give "excellent" results; (*c*) became less smooth in all of the procedures. They also show that the Type 4037 steel was very little improved with regard to cleanliness and was not improved at all with regard to smoothness by these various procedures. In every case, the embrittlement due to the pretreatment described was much greater than desired.

Since the procedure involving a 1-min treatment in hydrochloric acid with ultrasonics proved to be the most desirable in the first series of experiments, certain modifications of this procedure were investigated in Expt. 12-16, presented in Table V.

It was shown that a nitric acid treatment, after a hydrochloric acid treatment similar to that used in Expt. 11, did not change the degree of cleanliness of the Type 1062 steel but decreased its smoothness. On the other hand, it did improve the cleanliness of the Type 4037 steel while decreasing its smoothness.

It was shown in Expt. 13 that the addition of copper sulfate (copper coat), nitric acid, and hydrochloric acid with ultrasonics treatments after a hydrochloric acid treatment similar to that used in Expt.

Table IV. Surface conditions and degree of embrittlement of Type 1062 and 4037 steel pins after pretreatments

Expt. No.	Cleaning treatment	Steel type	Surface condition: Cleanliness	Surface condition: Smoothness	Degree of embrittlement: Angle of bend at fracture (°)	Degree of embrittlement: Proportion of pins fractured (%)
6	None	1062	Poor −	Good +	80-90	10
		4037	Poor −	Good +	45-115	83
7	H_2SO_4 (3 min), rinse.	1062	Poor −	Poor	8-35	100
		4037	Poor −	Poor	50-84	92
8	H_2SO_4 (3 min) with ultrasonics, rinse.	1062	Poor	Fair	45-78	100
		4037	Poor	Fair	58-119	75
9	HCl (3 min), rinse.	1062	Fair	Good	65-120	50
		4037	Poor	Good	30-129	87
10	HCl (3 min) with ultrasonics, rinse.	1062	Good	Good	>135	100
		4037	Poor	Good	68-99	60
11	HCl (1 min) with ultrasonics, rinse.	1062	Good	Good	>135	100
		4037	Poor	Good	68-100	50

Table V. Surface conditions of Type 1062 and 4037 steels after pretreatments (the angle of bend of 135 deg was reached without fracture in all cases)

Expt. No.	Cleaning treatments	Steel type	Surface condition after treatment: Cleanliness	Surface condition after treatment: Smoothness
12	HCl (1 min) with ultrasonics, rinse, HNO_3 (5 min), rinse, ultrasonic water rinse.	1062	Good	Poor −
		4037	Good	Poor −
13	HCl (1 min) with ultrasonics, rinse, copper coat, rinse, HNO_3 (1 min), rinse, ultrasonic water rinse, HCl (1 min) with ultrasonics, rinse.	1062	Good +	Poor −
		4037	Good +	Poor −
14	HCl (1 min) with ultrasonics, rinse, HNO_3-CH_3COOH (A) (3 min), rinse, ultrasonic water rinse.	1062	Excellent	Excellent
		4037	Excellent	Good
15	HCl (1 min) with ultrasonics, rinse, copper coat, rinse, HNO_3-CH_3COOH (A) (1 min), rinse, ultrasonic water rinse, HCl (1 min) with ultrasonics, rinse.	1062	Excellent	Excellent
		4037	Excellent	Excellent
16	HCl (1 min) with ultrasonics, rinse, copper coat, rinse, HNO_3-CH_3COOH (A) (1 min), rinse, air-water blast.	1062	Excellent	Excellent
		4037	Excellent	Excellent

11 made a definite improvement in the cleanliness of both steels while decreasing their smoothness. It was shown in Expt. 14 that the use of the HNO_3-CH_3COOH(A) treatment instead of the nitric acid treatment used in Expt. 12 produced "excellent" smoothness and surface cleanliness on the Type 1062 steel, and "excellent" surface cleanliness and only "good" smoothness on the Type 4037 steel. It should be noted that surfaces having "excellent" smoothness sometimes had very small, shallow indentations. However, these had none of the sharp edges that are particularly undesirable in plating.

It was shown in Expt. 15 that the addition of a copper coat treatment before the HNO_3-CH_3COOH(A) used in Expt. 14, and of a final hydrochloric acid treatment, produced "excellent" smoothness and cleanliness in both types of steel. It was shown in Expt. 16 that an air-water blast treatment may be used to replace the last two ultrasonic treatments used in Expt. 15.

Having completed the experiments in which it was found possible for pins of Types 1062 and 4037 high-strength steels to be made substantially clean, smooth, and free from embrittlement, and having previously shown that it is possible to produce stable cadmium plating solutions (3), the next step was to pretreat and plate these steels with the one operation following immediately after the other. The results are shown in Table VI.

It was shown in Expt. 17 that Type 1062 pins had increased in embrittlement when they had been plated after being subjected to the pretreatment used in Expt. 11, which had given "excellent" freedom from embrittlement but only "good" cleanliness and smoothness when used alone. On the other hand, it was shown in Expt. 18 that both types of steel had excellent freedom from embrittlement when they were plated after being subjected to the pretreatment used in Expt. 15, which had given "excellent" cleanliness and smoothness as well as "excellent" freedom from embrittlement when used alone. This would indicate that pretreated pins must be "excellent" with regard to cleanliness and smoothness as well as with regard to freedom from embrittlement if they are to be free from embrittlement after cadmium plating.

The pretreatment and plating procedure used in Expt. 19 was as effective as that in Expt. 18 and was simpler to perform. The excellence of the procedure used in Expt. 19 was further demonstrated when pins of both types of steel, which already had been bent through 135° in the Hounsfield testing machine, were placed in a vise and bent almost through 180° without breaking. It is of interest to note that the copper coat used in Expt. 19 could be eliminated without any ill effects when treating Type 1062 pins, but not when treating Type 4037 pins.

Table VI. Effect of pretreating followed by plating in a stable bath, on the embrittlement of Type 1062 and 4037 steel pins

Expt. No.	Procedure	Steel type	Degree of embrittlement after plating: Angle of bend at fracture (°)	Degree of embrittlement after plating: Proportion of pins fractured (%)
17	HCl (1 min) with ultrasonics. rinse, plate.	1062 4037	65-122 *	77
18	HCl (1 min) with ultrasonics, rinse, copper coat. rinse, HNO_3-CH_3COOH (A) (1 min), rinse, ultrasonic water rinse, HCl (1 min) with ultrasonics, rinse. plate.	1062 4037	>135 >135	100 100
19	HCl (10 sec). rinse, copper coat, rinse, HNO_3-CH_3COOH (B) (5 min), rinse, ultrasonic water rinse. plate.	1062 4037	>135 >135	100 100

* Not sufficiently clean for plating.

Type 4340 Steel.—Geyer, Lawless, and Cohen (1) described a number of experiments in which cadmium had been plated on bars of this type of steel using ordinary cyanide, fluoborate, and sulfamate baths. The plating had been followed by baking at 375°F (190°C) for 23 hr and then by testing on constant-load creep-rupture machines. Even though baked, a considerable proportion of the specimens failed early in the test. In a considerable proportion of those that did not fail, the appearance of the plated cadmium was undesirable. There is reason to believe that their results would have been even less satisfactory if they had used as small a radius at the bottom of the notches of their test bars as was used in the present experiments.

When four bars of the Type 4340 steel were pretreated and cadmium plated by the procedure used in Expt. 19, but not baked, they were ruptured after 147, 480, 140, and 144 hr in the constant-load machine (Expts. 20, 21, 22, and 23). It is of interest to note that, when similar bars were pretreated and plated with cadmium by typical commercial procedures, they fractured before the testing machine was fully loaded.

To summarize, it has been shown in these experiments that Types 1062 and 4037 high-strength steels can be electroplated with cadmium without significant embrittlement when the following requirements are met:

1. The steel surface is made clean, smooth, and free from embrittlement by the procedure: HCl (10 sec), rinse, copper coat, rinse, HNO_3-CH_3COOH (B) (5 min), rinse, ultrasonic water rinse.
2. The prepared steel surface is electroplated in a stable bath such as that given in Table I.

All specimens pretreated and plated in this manner were excellent with regard to coating adherence and appearance.

It was found that the usefulness of Type 4340 high-strength steel was greatly increased after treatment by this same procedure. However, it is believed that further research is required before entirely satisfactory results can be obtained.

Discussion and Conclusion

In this research, it was demonstrated that embrittlement occurs during the pretreatment and the cadmium plating of a typical high strength steel by normal commercial procedures. A method was then developed for preventing the appreciable embrittlement of Type 1062 high-carbon steel and Type 4037 silicon-molybdenum steel during pretreatment. This procedure leaves the surface clean and free from minute sharp edges, which are highly undesirable. Finally, it was shown that no appreciable embrittlement is produced when a properly pretreated surface is plated in a stable cadmium plating bath that had been developed previously in this laboratory. Considerable progress also was made in preventing embrittlement from occurring in Type 4340 silicon-nickel-chromium-molybdenum steel during pretreatment and cadmium plating. It is suspected that the method developed would be equally successful with a wide variety of other somewhat similar steels.

The most important steps in the new pretreatment are as follows:

1. Removal of visible films of foreign materials from the steel surface. Hydrochloric acid was found to be particularly successful in removing oxides while keeping the metal dissolution at a low rate.

2. Treatment in copper sulfate solution to produce a very thin copper coating on the steel. This assists in producing and maintaining a smooth steel surface during the nitric-acetic acid treatment of Type 4037 and 4340 steels in particular.

3. Treatment with a nitric-acetic acid solution, which produces a smooth surface free from minute

Table VII. Elimination of significant embrittlement from pins of Type 1062 and 4037 steels

Expt. No.	Procedure	Steel type	Degree of embrittlement: Proportion of pins fractured (%)	Degree of embrittlement: Angle of bend at fracture (°)
24	Cathodic treatment in HCl.	1062	100	1 to 10
		4037	100	1 to 10
25	Cathodic treatment in HCl, copper coat, rinse, HNO_3-CH_3COOH (A) (1 min), rinse, ultrasonic water rinse.	1062	100	>135
		4037	100	>135
26	As in Expt. 24, followed by plating in stable bath.	1062	100	>135
		4037	100	>135

sharp edges, without materially increasing the temperature. It also removes the thin copper coating.

4. Use of ultrasonic vibration in conjunction with acid treatment and water rinsing. This greatly increases the efficiency of these processes. It was shown that air-water blasting to remove thick smut from the steel surface after acid treatment is almost as effective and much less expensive than ultrasonic treatment.

During this research, there has been an opportunity to study the merits of the bend and constant load methods of testing for embrittlement. The former method has these advantages: 1—a test specimen is more readily produced, 2—a single test can be performed in a few minutes rather than in a number of days, and 3—the testing machine requires less floor space and costs much less.

Treatment to Eliminate Significant Embrittlement Produced by Chemical and Electrochemical Processes, from Type 1062 and 4037 Steel Pins

Although the prevention of appreciable embrittlement in a high-strength steel during pretreatment and plating is of very great importance, the elimination of embrittlement already present in such a steel also is of considerable importance. Accordingly, an investigation was undertaken in the hope of developing a successful procedure for this in the case of pins of Type 1062 and 4037 steels.

During the experiments, pins of these two types of steel were severely embrittled by cathodic treatment in hydrochloric acid. Then pins embrittled in the same way were given a copper coat and treated in HNO_3-CH_3COOH(A) solution, which removed all significant embrittlement. Embrittled pins, after treatment in the above manner and then cadmium plating in a stable bath, still were not embrittled appreciably. These results are summarized in Table VII. It is interesting to note that the combination of copper coating and treatment in HNO_3-CH_3COOH solution is useful in both the prevention and elimination of embrittlement from these steels.

In the past, there has been considerable disagreement as to the nature of embrittlement in steels. The type of embrittlement dealt with in the present experiments appears to originate in a thin layer existing at the surface of the metal. When an embrittled test specimen is bent or stretched, a crack apparently is developed in this layer and spreads quickly through the specimen, resulting in fracture. This seems to be the most satisfactory explanation in view of the fact that the embrittlement can be eliminated by a treatment in a chemical solution.

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Any discussion of this paper will appear in a Discussion Section to be published in the Nov.-Dec. 1967 issue.

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