



# Standard Guide for Preparation of High-Carbon Steel for Electroplating<sup>1</sup>

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## 1. Scope

1.1 This guide is intended as an aid in establishing and maintaining a preparatory cycle for electroplating on high-carbon steel (Note 1) producing a minimum of hydrogen embrittlement and maximum adhesion of the electrodeposited metal. For the purpose of this guide, steels containing 0.35 % of carbon or more, and case-hardened low-carbon steel, are defined as high-carbon steels. There is no generally recognized definite carbon content dividing high from low-carbon steels for electroplating purposes.

NOTE 1—Electroplating of plain high-carbon steel introduced problems not found in similar operations on low-carbon steel. During the cleaning and electroplating cycle, high-carbon steel differs from low-carbon steel in regard to its greater tendency to become embrittled and the greater difficulty in obtaining maximum adhesion of the electrodeposit. The preparation of low-carbon steel for electroplating is covered in Practice B 183.

1.2 This guide does not apply to the electroplating of alloy steel. For methods of chromium electroplating directly on steel see Guide B 177.

1.3 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazards statement, see 3.1.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- B 177 Practice for Chromium Electroplating on Steel for Engineering Use<sup>2</sup>
- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating<sup>2</sup>
- B 849 Specification for Pre-Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement<sup>2</sup>
- B 850 Specification for Post-Coating Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement<sup>2</sup>

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 02.05.

## 3. Reagents

3.1 *Purity of Reagents*—All acids and chemicals used in this practice are technical grade. Acid solutions are based upon the following assay materials:

Hydrochloric acid (HCl) 31 mass %, density 1.16 g/mL

Nitric acid (HNO<sub>3</sub>) 67 mass %, density 1.40 g/mL

Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 93 mass %, density 1.83 g/mL

**Caution**—Dilute sulfuric acid by slowly adding it to the approximate amount of water required with rapid mixing. After cooling, bring the mixture to exact volume.

3.2 *Purity of Water*—Use ordinary industrial or potable water for preparing solutions and rinsing.

## 4. Nature of Steel

4.1 *Hardness*—High hardness is a major cause of cracking of the steel during or after electroplating. The recommended maximum hardness range for classes of products depends on their geometry and service requirements (Note 2). Parts hardened by heat treatment should be inspected before electroplating for the presence of cracks by a suitable method, such as magnetic or fluorescent powder inspection.

NOTE 2—Some examples of parts and Rockwell hardness ranges are as follows:

	Rockwell Hard- ness Range
Springs	C45 to C48
Spring washers	C45 to C53
Small instrument parts	C52 to C55
Parts to be chromium electroplated for engineering use	C57 to C62

4.2 *Hydrogen Embrittlement*—Difficulties resulting from hydrogen embrittlement increase with increasing hardness, whether produced by heat treatment or cold work. Difficulties, during or after electroplating of hardened high-carbon steel parts, may in some cases be minimized without material change in hardness by baking before final pretreatment. For a listing of such hydrogen embrittlement relief bake cycles, consult Specification B 850.

4.3 *Surface Oxidation*—In order that subsequent treatments be facilitated, every reasonable precaution should be taken throughout the processing to limit oxidation or scale formation. In particular cases pre-electroplating with copper to a minimum thickness of 13 μm may assist in maintaining a preferred surface through the heat treatment. A nonoxidizing atmosphere

should be maintained in the furnace. This copper shall be removed prior to the regular electroplating cycle. Care should be used in oil-quenching parts heat treated in a salt bath, to prevent the charring effect that can be caused by salt-bath drag-out. Proper lead-bath quenching results in only slight oxidation.

4.4 *Steel Quality*—The quality of the steel should be characteristic of the requirement of the product and the electroplating operation. The steel should be free of injurious surface defects, and of at least average cleanliness.

## 5. Preparation of Steel, General

5.1 *Preparatory Treatments*—A wide variety of surface conditions are encountered in high-carbon steel articles to be electroplated. The surface may require the removal of one or more of the following contaminants: grease, oil or drawing compounds, burned-in oil scale, light to heavy treatment scale, permeable oxide films, emery and fine steel particles resulting from the grinding operation. The removal of such contaminants is accomplished by one or more of the following pretreatment procedures where applicable:

5.1.1 Substantial removal of oil, grease, and caked-on dirt by precleaning before the part enters the electroplating cycle (applicable in all cases).

5.1.2 Mechanical treatment of the surface by tumbling, sand or grit blasting, vapor blasting, or grinding (optional).

5.1.3 Final and complete anodic cleaning in an electrolytic alkali cleaner.

5.1.4 Acid treatment in HCl to remove the last trace of oxide and scale. This should be avoided for spring temper and case-hardened parts. This treatment also removes residual traces of lead that may be present following proper lead-bath quenching.

5.1.5 Smut removal by cyanide dipping or by anodic treatment in cyanide or alkali.

5.1.6 Final preparation for electroplating may be accomplished by an anodic etching treatment in H<sub>2</sub>SO<sub>4</sub> (used whenever possible in the interest of high yield and adhesion).

5.1.7 Conditioning of the surface to be electroplated may be accomplished, where necessary for the electroplating process, by a short dip or rinse in a solution equivalent to the electroplating solution without its metallic content.

5.2 *Rinsing*—Inadequate rinsing after each solution treatment step is the recognized cause of a large portion of electroplating difficulties. Not enough rinsing is characteristic of most pretreatment cycles.

5.3 *Pretreatment Time*—All processing steps involving hydrogen generation must be designed to operate for a minimum length of time, to avoid hydrogen embrittlement of the high-carbon steel.

5.4 *Control*—All pretreatment steps should be carried out with solutions that are maintained in good working condition by control of composition and contaminants, and used under conditions of time, temperature and current density specified to meet the requirement of the work being processed.

5.5 *Pretreatment Cycle Design*—Depending upon the requirements for the particular high-carbon steel parts to be electroplated, a minimum cycle should be selected from the general steps listed in 5.1. Different classes of materials require

selected process steps combined into pretreatment cycles of greater or less complexity according to the condition and properties of the material. The minimum number of steps necessary to accomplish the electroplating satisfactorily is recommended.

## 6. Preliminary Pretreatment Procedures

6.1 *Application*—Degreasing and mechanical surface treatment are necessary only where the high-carbon steel parts are contaminated to such an extent that otherwise the burden imposed on the pretreatment cycle would impair its efficiency, increase its complexity, and tend to prevent the attainment of the required quality of the deposit. The overall cost of the electroplating process is usually reduced by using the preliminary treatments where applicable. Oil, grease, dirt, drawing compounds, burnt-in oil, heavy scale, and emery and steel particles are typical of the gross contaminants encountered.

6.2 *Precleaning*—Solvent-degreasing with clean solvent, spray-washing, or emulsion-cleaning, followed by electrolytic or soak-alkali cleaners are recommended. The former types are preferred to reduce the burden on the alkali treatments. Soak-alkali cleaning is usual for parts that are to be barrel electroplated. Electrolytic cleaning should always be anodic where the control of embrittlement is a problem.

6.3 *Stress Relief Treatment*—It is recommended that hardened high-carbon steel parts receive a stress-relief bake before the parts are mechanically pretreated or enter the final pretreatment cycle, or both. For a listing of typical stress-relief bakes, consult Specification B 849.

6.4 *Mechanical Treatment*—The purpose of mechanical treatment is to reduce subsequent acid pickling to a minimum. Where mechanical treatment has been accomplished with precision, it is sometimes possible to eliminate acid pickling entirely, thus improving the control of hydrogen embrittlement. When required, mechanical treatment of small parts is best effected by tumbling. All scaled and nearly all oil-quenched materials require mechanical cleaning such as by tumbling with or without abrasive, or by sand, grit, or vapor blasting. These operations should be carried out so as to avoid severe roughening of the surface with accompanying notch effect. One resorts to grinding in certain cases where the surface smoothness or dimensions of the parts are of critical importance, for example, in chromium electroplating for engineering use.

## 7. Final Pretreatment Procedures

7.1 *Application*—Final cleaning, oxide removal, and anodic acid treatment are fundamental steps required for preparing high-carbon steel for electroplating. These pretreatment steps are designed to assist in the control of hydrogen embrittlement and in securing the maximum adhesion of the electroplated coating.

### 7.2 *Electrolytic Anodic Cleaning:*

7.2.1 All work, except work to be barrel electroplated, should preferably be cleaned in an electrolytic anodic alkaline cleaner. Anodic cleaning is recommended to avoid hydrogen embrittlement that is likely to result from cathodic cleaning. An exception is barrel work which, because of the work size, is preferably cleaned by soaking or tumbling in an alkaline

cleaning solution without the use of current.

7.2.2 The purpose of this cleaning step is to remove completely the last traces of contaminants. In all cases it should be preceded by heavy-duty precleaning as covered in 6.2.

7.2.3 The electrolytic anodic cleaner should be used at a temperature of 90°C or higher, and at a current density of 5 A/dm<sup>2</sup> or higher, in order that the required degree of cleanliness be obtained in a time period not exceeding 2 min.

7.2.4 On removal from the cleaner, the work should be thoroughly rinsed, first with water warmed to 50°C, and then in a cold-water spray at room temperature, prior to the acid dip.

### 7.3 Rinsing:

7.3.1 The most thorough fresh-water rinsing operation possible is mandatory after each processing step if the best results in electroplating high-carbon steel are to be obtained. The purpose of rinsing is to eliminate drag-over by complete removal of the preceding solution from the surface of the work. Many existing commercial operations are characterized by inadequate rinsing.

7.3.2 Warm to hot rinses should be used following alkaline solutions or where the subsequent processing solution is hot. The rinse temperature should not be so high as to induce drying of the steel surface between processing steps. Room temperature rinses are suitable for use following acid solutions where the solution in the next processing step is cold. In no case should very cold water be used for rinsing.

7.3.3 The recommended rinsing practice includes the use of an immersion rinse, always followed by a spray rinse of fresh water at the required temperature. Not using a spray rinsing is an invitation to trouble in the electroplating of high-carbon steel.

7.4 *Hydrochloric Acid Treatment*—The purpose of the HCl treatment is to remove completely the last trace of oxide from the surface of the high-carbon steel. The intensity of the HCl treatment should be held to the minimum required by the nature and amount of oxide present. The use of H<sub>2</sub>SO<sub>4</sub> instead of HCl is not recommended for descaling high-carbon steel because of its smut-forming tendency, in spite of the somewhat lowered tendency to rusting of H<sub>2</sub>SO<sub>4</sub>-treated surfaces. The addition of wetting agents to the HCl solution is not recommended. Care and caution must be exercised in the use of inhibitors where they are required, because they sometimes interfere with adhesion. Inhibitors are of benefit only in special cases where surface finish and dimensions are of prime importance.

7.5 *Treatment for Smut Removal*—When the HCl treatment of the high-carbon steel results in the presence of smut, the smut must be removed before the surface is electroplated. Light oxides formed on exposure to air after acid treatment must likewise be removed. This can be done by an anodic cyanide or alkaline treatment. Air-formed oxide, if not too heavy, can be removed by a cyanide dip after the rinse following the acid treatment. A concentration of 22 g/L of NaCN is sufficient for the cyanide dip. Where a severe smut condition exists, it can be eliminated by a ½ to 1-min anodic treatment at 1.5 to 2 A/dm<sup>2</sup> in a solution of a NaCN of the noncritical concentration of 45 g/L used at room temperature. An alternative treatment for a somewhat lighter smut condition

is electrolytic anodic treatment in the noncyanide alkaline cleaning solution (6.3) above 70°C, for 15 to 30 s at 2.5 to 5 A/dm<sup>2</sup>. The current density is not critical.

### 7.6 Anodic Acid Etching:

7.6.1 The use of an anodic acid etch and subsequent rinse as final steps in the preparation for electroplating of high-carbon steel is of importance in securing adhesion. Without such an anodic treatment, poor adhesion may occur. The anodic acid treatment is capable of removing a small amount of smut formed by the preceding HCl treatment; more substantial amounts of smut should be removed according to the procedures described in 6.5.

7.6.2 A 150 to 600 mL/L H<sub>2</sub>SO<sub>4</sub> solution used at a temperature of not more than 30°C, and preferably below 25°C, is effective for anodic etching of high-carbon steel. See **Caution** in 3.1. The addition of 125 g/L of Na<sub>2</sub>SO<sub>4</sub> (based on the anhydrous salt) is of benefit for many steel grades. Anodic treatment in this solution for a time usually not exceeding 1 min at a current density of 16 A/dm<sup>2</sup> (range of 10 to 43 A/dm<sup>2</sup>) is sufficient. A high acid content, high current density, and low temperature (with reference to the ranges specified) will minimize the attack on the basis metal and produce a smoother surface. This H<sub>2</sub>SO<sub>4</sub> solution is very stable and not affected appreciably by iron build up. Besides securing adhesion of the subsequent electrodeposit, it improves the uniformity of the deposit and reduces hydrogen embrittlement.

7.6.3 A dip for 5 to 10 s in a 55 mL/L HNO<sub>3</sub> solution, followed by rinsing and anodic cyanide treatment for smut removal, has in certain cases been found effective. Although nonelectrolytic, the HNO<sub>3</sub> treatment requires an additional electrolytic cyanide treatment before electroplating.

### 7.7 Electropolishing:

7.7.1 Electropolishing is used to remove highly stressed metal and metal debris from the surface of cold-worked steel, thereby improving the bond strength and corrosion resistance of electroplated coatings. It accomplishes this function without the tendency to form smut, which may result from anodic etching. Because it does not etch the steel, it is preferred by some electroplaters to anodic etching for preparing steel surfaces for decorative electroplating. Proprietary mixtures of phosphoric and sulfuric acids<sup>3</sup> for electropolishing are of special interest in view of their ability to remove smut from cast iron surfaces. The addition of chromic acid to sulfuric-phosphoric acid mixtures<sup>4</sup> further provides a surface passivation that is beneficial in preventing rusting during transfer or during temporary storage of steel prior to electroplating.

7.7.2 An activating treatment after electropolishing can be beneficial to subsequent electroplating. An example of a suitable activating treatment is anodic cleaning followed by acid dipping. Electropolishing can be used in addition to or instead of anodic etching.

7.7.3 Electropolishing of fine-grained steel provides a uniform, smooth finish. Electropolishing of a coarse-grained structure (No. 6 or coarser) results in less smoothness. In either case, however, a word of caution must be interjected, namely

<sup>3</sup> U.S. Patent 2,334,699.

<sup>4</sup> U.S. Patents 2,366,712 and 2,338,321.

that any seams, voids, stringers, and other surface defects can be damaging to the appearance of the electroplated coating. Yet, the removal of sharp edges or scratches and the removal of nonmetallic inclusions in seams and stringers can reduce the harmful effect of these defects on the corrosion resistance of the electroplated steel.

## 8. Electroplating Procedures

8.1 Standard electroplating procedures can be used on high-carbon steel when the proper preparatory steps described in Sections 6 and 7 have been selected and followed. The conditions of use of the electroplating bath should assure a minimum evolution of hydrogen at the cathode surface (highest cathode current efficiency). The use of the minimum length of time in each step of preparation and electroplating is recommended. The material should be handled with the minimum number of steps consistent with proper treatment.

8.2 The electrodeposition of tin and cadmium on high-carbon steel is easier to accomplish than that of zinc. Electrodeposition of nickel and chromium is not difficult, if the recommended practice is followed. Low internal stress of the deposit is desirable.

8.3 Springs and similar materials should not be electroplated while subject to externally applied stress.

## 9. Heat Treatment After Electroplating

9.1 *Application*—One purpose of the special preparatory treatments of high-carbon steel (Sections 6 and 7) is to minimize hydrogen embrittlement. In most commercial production it is necessary to bake the electroplated work for final

embrittlement relief. With springs and similar materials, precautions should be observed to avoid flexing the articles before they are baked.

9.2 *Procedure*—The hydrogen may be largely removed and the physical properties of the steel substantially restored by heating, for example, for 1 to 5 h at temperature in an oven maintained at a temperature of 150 to 260°C, the temperature and length of treatment depending on the severity of embrittlement, the cross section of the article, the requirements of the steel, and the kind and thickness of the electrodeposited coatings. The baking should be done as soon as possible after electroplating, and before any supplementary chemical treatment of the electroplated surfaces. The best time and temperature in some cases must be established experimentally. A temperature not exceeding 205°C is recommended for zinc- or cadmium-electroplated articles. A lower temperature may be required if the coating is to be given a subsequent chemical treatment.

## 10. Test Methods

10.1 *Adhesion*—No universally satisfactory nondestructive test for adhesion is known. Poor adhesion may be revealed during grinding operations.

10.2 *Embrittlement*—The test for the effectiveness of the procedures used to control embrittlement lies in the subsequent service use of the material. Applicable control tests for embrittlement can be selected by analogy to the particular service in which the high-carbon steel article being processed will be used.

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