



Standard Practice for Preparation of Nickel for Electroplating with Nickel¹

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1. Scope

1.1 This practice summarizes well-known, generally practical methods for producing adherent electrodeposits of nickel on nickel.

1.2 Electrodeposits of nickel on nickel are produced, for example, to improve the performance of decorative coatings, to reclaim electroplated parts that are defective, and to resume nickel electroplating after interruptions in processing. Interruptions may be deliberate, for example, to machine the electrodeposit at an intermediate stage in the electrodeposition of thick nickel coatings. The interruptions may be unintentional, for example, resulting from equipment and power failures.

1.3 To ensure good adhesion of nickel to nickel, precautions should be taken to avoid biopolar effects during nickel electroplating. This is of particular importance in return-type automatic plating machines where one rack follows another rack closely. Bipolar effects can be avoided by making the racks cathodic while they are entering or leaving the nickel tank. Separate current control on entry and exit stations is desirable.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, 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.*

2. Types of Nickel

2.1 The types of nickel for which an overplate of nickel may be desired are dull nickel, semi-bright nickel, bright nickel, and nickel strike. Variations in these types may possibly require special handling.

¹ This practice is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.02 on Pre Treatment.

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- 2.2 Surface conditions of the nickel may vary as follows:
- 2.2.1 Freshly electroplated surfaces that are still wet with electroplating solution or rinse water (see 5.1),
 - 2.2.2 Freshly electroplated surfaces that have been allowed to dry (see 5.2),
 - 2.2.3 Buffed, polished, or machine-ground surfaces (see 5.3), and
 - 2.2.4 Surfaces that have been given a reverse-current treatment in an alkaline solution for cleaning or possibly stripping an overplate of chromium (see 5.4).

3. Cleaning

3.1 The following cleaning treatments may be used for all conditions and types of electrodeposited nickel. The choice of the procedure will be governed largely by the condition of the surface.

3.1.1 *Degreasing*—Degreasing is used to remove the bulk of grease, oil, and buffing compounds that may be present on the surface. The cleaning may be effected with vapor degreasing, organic solvents, emulsion cleaners, or soak cleaner.

3.1.2 *Electrolytic Alkaline Cleaning*—Removal of final traces of dirt, grease, and oil is accomplished best with electrolytic alkaline cleaning. The solution may be either a proprietary cleaner or a formulated one. Since a nickel surface forms an oxide coating if treated anodically in an alkaline solution, this condition must be altered in subsequent steps if it cannot be avoided.

4. Activating

4.1 The procedure used for etching or activating the nickel surface usually determines the soundness of the adhesion. The choice of the procedure may be governed by the condition of the surface and possibly the type of nickel. The milder etching treatment should be used in the case of highly finished surfaces, but it may result in sacrificing maximum adhesion. The thickness of the nickel may militate against the use of certain etching procedures, and therefore the thickness removed is indicated for each procedure described in 4.2 to 4.8.

4.2 *Anodic Treatment in Concentrated Sulfuric Acid*—(Nickel removed nil). A70 mass % sulfuric acid solution containing 661 mL of concentrated, 96 mass % sulfuric acid

(density 1.83 g/mL) diluted to 1 L may be used for activating the nickel surface provided the temperature of the solution is not over 30°C (see **Warning**). When the initial mixture cools, dilute to exact volume. The time of treatment should be about 1 min at a current density of 10 A/dm². At this current density the nickel normally goes passive and a bright surface becomes only slightly dull. This type of passivity is removed by subsequent rinsing in water. (**Warning**—Slowly add the sulfuric acid with rapid stirring to the approximate amount of water required.)

4.3 Anodic Etching in Sulfuric Acid—(Nickel removed approximately 1.3 μm.) A25 mass % sulfuric acid solution, containing 166 mL of concentrated, 96 mass % sulfuric acid (density 1.83 g/mL), diluted to 1 L is used for this anodic etching treatment in which the nickel surface is first etched at a low current density of 2 A/dm² for 10 min and then made passive at 20 A/dm² for 2 min and finally cathodic for 2 or 3 s at 20 A/dm². See **Warning** in 4.2. The temperature of the solution should be kept below 25°C. This treatment results in excellent adhesion, but the amount of etching makes it less desirable for a highly finished surface.

4.4 Anodic Etching in Watts-Type Bath—(Nickel removed approximately 4 μm.) This procedure employs an anodic treatment in a low-pH Watts bath for 3 min at 1 A/dm² (10 A/ft²), followed by cathodic treatment for 3 to 6 min at 3 A/dm² (30 A/ft²). The composition of the solution is 240 to 300 g/L nickel sulfate (NiSO₄ · 7H₂O), 40 to 60 g/L nickel chloride (NiCl₂ · 6H₂O), and 25 to 40 g/L boric acid. It is operated at a temperature between 25 and 50°C and at a pH between 1.5 and 2.0. An additional bath is not required if a means of reversing the current is available. The amount of etching obtained is sufficient to dull a bright surface, and there is danger of bath contamination from bare areas.

4.5 Acid-Nickel Chloride Treatment—(Nickel removed approximately 1.3 μm.) This procedure employs an anodic treatment followed by a cathodic treatment in a low-pH nickel chloride solution. The composition of the solution is 240 g/L of nickel chloride (NiCl₂ · 6H₂O) and 31 mL of concentrated, 37 mass % hydrochloric acid (density 1.16 g/mL). The normal procedure is to make the work anodic for 2 min at 3 A/dm² and then cathodic for 6 min at the same current density. Where it is not possible to reverse the current in the same tank, the two steps may be carried out in separate tanks. The latter arrangement may be more practical commercially. This treatment produces a dull finish on a bright surface, but the etching is not sufficiently drastic to preclude finishing with bright nickel deposit.

NOTE 1—Nickel anode materials containing greater than 0.01 mass % sulfur are not recommended for use in acid nickel strike baths operating at pH 0.5, or lower, to avoid oxidation of sulfides by hydrochloric acid.

4.6 Etching by Acid Immersion—(Nickel removed approximately 1.3 μm.) Adequate etching may be obtained on some nickel surfaces by a short dip at room temperature in a solution of either 500 mL of concentrated 37 mass % hydrochloric acid (density 1.18 g/mL) diluted to 1 L, or 150 mL of concentrated

96 mass % sulfuric acid (density 1.83 g/mL) diluted to 1 L. See 4.2. The length of the immersion required may vary from 10 s to 1 min.

4.7 Electropolishing Treatment—(Nickel removed approximately 1.3 μm.) This procedure is commonly employed on rejects that have been repolished to remove the defective area. The electropolishing solution commonly used consists of a mixture of 150 mL of 96 mass % sulfuric acid (density 1.83 g/mL) and 630 mL of 85 mass % phosphoric acid (density 1.69 g/mL) diluted to 1 L. See **Warning** in 4.2. Temperature of solution ranges from 45 to 55°C. The work is made anodic at current densities from 15 to 20 A/dm². The electropolishing treatment is usually applied for 2 to 15 min. The cathodes may be electrolytic nickel strip. Subsequent alkaline cleaning and an acid dip are normally used before electroplating.

4.8 Cathodic Treatment—(Nickel removed nil.) These procedures are recommended where the nickel surface has not been severely passivated. Prior cleaning may be required, such as alkaline soak cleaning or electrocleaning, or both. If electrocleaning is employed, only cathodic current should be used. In the following formulations, 96 mass % sulfuric acid with a density of 1.83 g/mL, and 37 mass % hydrochloric acid with a density of 1.16 g/mL, are used.

4.8.1 Cathodic Treatment in Sulfuric Acid:

Sulfuric acid	30 to 100 mL
Water	to 1 L
Temperature	ambient to 45°C
Current density	1 A/dm ²
Anodes	pure lead
Time	30 s to 5 min

4.8.2 Cathodic Treatment in Hydrochloric Acid:

Hydrochloric acid	100 to 300 mL
Water	to 1 L
Temperature	ambient
Current density	1 A/dm ²
Anodes	electrolytic nickel strip or nickel bar anodes
Time	30 s to 5 min

NOTE 2—In some instances, the use of 2 g/L of ammonium bifluoride (NH₄ · HF) has been found to be beneficial in either of the above formulations.

NOTE 3—Proprietary acid salt formulations may be used with the supplier's recommendations.

5. Electroplating Procedures

5.1 Wet Freshly Electroplated Surfaces—Nickel surfaces that are still wet with electroplating solution can sometimes be placed directly in the subsequent nickel electroplating bath without any special treatment. If the solutions are not compatible, a water rinse may be used between nickel baths. This procedure will normally give good adhesion with Watts-type nickel and certain semi-bright nickels but may give poor adhesion with bright nickels. A mild etch, such as a short

immersion in a dilute acid solution, may be required for some semi-bright and bright nickel surfaces.

5.2 Dry Freshly Electroplated Surfaces—Nickel surfaces that have been allowed to dry following the rinse from the electroplating bath may only require an activation treatment. However, if there is a possibility of a trace of oil or grease on the surface, a cathodic alkaline cleaner should be used first. A short immersion in a dilute acid solution should be adequate to activate most nickel surfaces, but certain bright nickels may require more drastic activation.

5.3 Polished, Buffed, or Machine Ground Surface—These nickel surfaces can usually be treated alike for nickel electroplating, although the buffed surface will require more drastic degreasing treatment and alkaline cleaning. For decorative surfaces, a mild etch, such as a short immersion in a dilute acid or a short anodic treatment in sulfuric acid, should be adequate to assure good adhesion.

5.4 Anodic Alkaline Treated Surface—The oxide film on a nickel surface from anodic treatment in an alkaline solution must be removed by a suitable activation treatment before the nickel surface can be electroplated adherently with nickel. An acid dip or a mild anodic etch in sulfuric acid is usually not adequate. A heavy anodic etch in sulfuric acid, an electropolishing treatment, a low-pH nickel bath, or the acid-nickel chloride treatment will normally be required to provide a sound deposit. A wipe on a buffing wheel will also serve to remove the oxide film.

5.5 Rejects with Chromium Electroplate—On parts requiring the removal of chromium for reprocessing, use of an anodic alkaline chromium strip should preferably be avoided. Acid stripping should be used. If an anodic alkaline strip is used, then an anodic acid etch is absolutely necessary.

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