Endorsed by American Electroplaters' Society Endorsed by National Association of Metal Finishers

Standard Guide for Cleaning Metals Prior to Electroplating¹

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INTRODUCTION

This guide is intended to illustrate general principles of cleaning prior to electroplating. It is not meant to apply to every specific application. In specific cases, cleaning practice may depart from the general principles given in this guide.

1. Scope

- 1.1 This guide describes the procedure for cleaning metal surfaces to obtain good adhesion of electrodeposited metals. The degree of cleanliness required for metals to be electroplated is greater than for most other finishes. Methods of removal of heat-treat or mill scale are not included in these methods, because they are covered in practices referring to specific metals. It should also be understood that while these procedures are broadly applicable, particular substrates may require certain specific cleaning procedures.
- 1.2 Adequate cleaning requires a proper combination of cleaning procedures. The choice of these procedures must be based on a knowledge of the metals to be cleaned and of the soils to be removed. Because most experience and knowledge in cleaning have been obtained by suppliers of proprietary processes and formulations, these sources should be consulted before setting up a cleaning process.
- 1.3 A treatment to remove tarnish, light rust, fingerprints, or oxides is usually provided before immersion of the piece in the electroplating tank. This treatment activates the metal and is usually accomplished in acid baths which also serve to neutralize the residual alkaline film from alkaline cleaning. Alkaline chelated derusting and cleaning solutions, alone or with sodium cyanide, used as a soak or electrocleaner, are often preferred before electroplating on ferrous alloys.
- 1.4 Invariably several stages are necessary to provide adequate cleaning. These stages are discussed in three parts:

Part I—Precleaning (use of a solvent, emulsion, or alkaline spray) to remove the bulk of the soil.

Part II—Intermediate (alkaline) cleaning.

Part III—Final electrocleaning, to remove trace solids and especially adherent impurities.

Part IV—Trouble shooting.

Often, depending largely on the amount and type of soil on the workpieces as received, one or more of these stages may be eliminated or modified. Usually, even with light soils, it is advisable to retain multistage cleaning, thereby increasing the life and efficiency of the cleaning solutions.

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. (For more specific safety precautionary statements see Sections 11 and 16.)

2. Significance and Use

2.1 The performance and quality of electroplated articles depend upon the surface cleanliness and condition. Various metals are electroplated for decorative or engineering finishes. The common electroplates applied are usually copper, nickel, and chromium for decorative and functional uses. Electroplated articles are used in many industries such as the marine, automotive, plumbing fixtures, and appliance industries.

3. Nature of the Soil

- 3.1 Some of the soils commonly encountered in electroplating are:
- 3.1.1 Solid buffing compounds containing waxes, fatty acids, and abrasives.
 - 3.1.2 Liquid buffing compounds.
- 3.1.3 Drawing and stamping compounds including those containing fillers (pigments).

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- 3.1.4 Machining oils.
- 3.1.5 Rust-preventive slushing oils or greases.
- 3.1.6 Electroplater's stop-off residues.
- 3.1.7 Fingerprints.
- 3.1.8 Dry dirt from storage or dry pickling smut formed during derusting by pickling.
- 3.1.9 Rust or oxide scales, especially admixed with oil, including heat-treat scales after oil quenching.
 - 3.1.10 Phosphate coating with or without lubricant.
- 3.1.11 Smut resulting from improper vapor degreasing of heavily buffed work.
- 3.1.12 Smut resulting from annealing parts without precleaning between drawing operations.
 - 3.1.13 Heat-treating salts, with or without quenching oils.
- 3.2 Consideration should be given to control of the soil. For example, efforts should be made to avoid overbuffing, leaving excessive compound on the work, or aging of the compound on the part before cleaning. Substitution of liquid for solid buffing compound, if work permits, often gives easier cleaning, if properly applied, but may require use of a different type of cleaner. Drawing compounds with polymerizing oils or white lead pigment are to be avoided because of difficulty in cleaning. Additives for lubricating and sulfurized cutting oils are chosen for their ability to adhebe tenaciously and are difficult to remove. Prolonged storage or drying of emulsion drawing compounds after metal working should be avoided so that slimy water-in-oil emulsions do not form. In-process cleaning or even a hot-water flush before storage is helpful. Emulsion machining lubricants (soluble oils) should be used in place of sulfurized cutting oils if operations permit. Lowerviscosity machining and rust-preventive oils are more easily removed. Stop-off materials, when used, should be applied carefully in order to avoid contaminating significant surfaces. The use of clean gloves should be mandatory after buffing or polishing to avoid fingerprints on the work. Airborne contaminants can be avoided by using covers over stored work. It is desirable to perform a cleaning operation as soon as possible after metal forming, polishing, or buffing to reduce the demands on subsequent cleaning operations, because many soils are more easily removed when fresh.

4. Metal

4.1 The properties of the metal and the method of fabrication and handling of parts play a role in cleaning. The softness and surface finish of the metal are factors in selecting handling methods. The chemical activity of the metal is an important and determining factor in cleaner selection. Aluminum requires care to avoid overetching in alkaline cleaners; both aluminum and zinc are sensitive to pitting attack, zinc and brass to tarnishing. Zinc die castings have surfaces that require special care because of sensitivity to attack by cleaning solutions. If possible, design of parts should avoid small indentations that tend to trap solid particles or buffing compositions. With die castings, care must be exercised to avoid cutting through the surface by excessive buffing. The subsurface is usually more sensitive than the" skin" of the casting. Some surface defects may not show up until cleaning and electroplating cycles are completed.

5. Cleaner

- 5.1 It is essential that proper cleaners and operational conditions be selected. Attention should be given to proper procurement since, even in the same category, not all cleaners are equally effective. A cleaner may be very effective for one group of soils, yet poor for other soils. This is true of electrocleaners as well as soak or spray cleaners. Soil, type of water, available time, rinsing facilities, type of metal, heating, and agitation available, facilities for disposal of cleaner, and type of personnel and equipment all influence the selection of cleaners. Obviously, economics must be considered but an initial or per pound cost must be balanced against other factors.
- 5.2 Cleaners do not work effectively indefinitely. The effective life of a cleaning bath must be estimated and baths discarded when exhausted. Bath life is influenced by some of the factors mentioned above as well as by the volume of work processed. The concentration of the cleaner should be controlled by analysis at regular intervals.

6. Rinses

6.1 Water hardness, acidity or alkalinity, and impurities are important factors in rinsing (1).² Distilled or demineralized water is preferred where impurities in rinse water must be kept to a minimum. Boiler condensate may also be used advantageously. If the plant conditions water for acidity or alkalinity care must be taken to be sure the solids content is not too high (Note 1). Impurities derived from processing cannot be ignored; that is, rinse waters must be changed frequently or overflowed continuously (Note 2). Counterflowing rinses are a distinct advantage in obtaining good rinsing with economical use of water.

Note 1—Boiler waters which contain cationic corrosion inhibitors may be quite detrimental to the plating process.

Note 2—Floating oil on water can cause poor adhesion.

7. Equipment

7.1 It is important to provide enough room in the plant for an adequate cleaning cycle. A discussion of equipment is beyond the scope of this practice (2, 3).

8. Criteria of Cleanliness

- 8.1 This subject has been treated exhaustively in the literature (4). The atomizer test is the most sensitive one, but the water-break test is most commonly used. This involves visual observation after a final rinse in clear, cool water. A continuous sheet of water on the part usually indicates a clean surface. (Certain precious-metal surfaces, such as gold, may exhibit water break, even though clean.) Some experience is necessary to judge the appearance of a break in the film of water. A specific drainage time, about 30 s, should be used before observation.
- 8.2 A dip in clean, dilute acid and reexamination are desirable to avoid false water-film continuity due to adsorbed soaps. Other methods, including electroplating and testing of

² The boldface numbers in parentheses refer to the reports and papers appearing in the list of references at the end of this practice.

the electroplate, should be used occasionally to confirm visual examination. (One procedure involves scrubbing with pumice and then comparing the surface produced by this method with that produced under production conditions.)

PART I—PRECLEANING

9. Purpose

9.1 Precleaning is designed to remove a large excess of soil, especially deposits of buffing compound or grease. It is also useful in reducing the viscosity of waxes and heavy oils, to enable later cleaning stages to be more effective, or to surround fingerprints and dry dust with an oily matrix to facilitate removal by alkaline cleaners.

10. Types

10.1 Cold solvent, vapor degreasing, emulsifiable solvent, solvent emulsion spray, invert-type emulsion cleaners, or hot alkaline spray with or without solvent emulsion can be used (5).

10.2 Cold Solvent (6)—Mineral spirits; trichloroethylene; perchloroethylene; 1,1,1,-trichloroethane (methylchloroform); methylene chloride; or trichlorotrifluoroethane can be used for cold cleaning. Combining these with hand brushing is excellent but does not lend itself to production conditions. On the other hand, simple dipping in solvent is frequently ineffective. The chlorinated solvents are very effective for many soils, but not as effective in removing soap-based or other solvent-insoluble soils. Before electroplating, cold cleaning with solvents must be followed by additional cleaning such as alkaline cleaning to remove slight oily residues.

10.3 Vapor Degreasing (7)—Trichloroethylene and, to a lesser extent, perchloroethylene, trichlorotrifluoroethane, and methylene chloride are used for vapor degreasing. In vapor degreasing, the work is usually sprayed with clean solvent or given a thorough immersion in boiling or warm solvent for mechanical removal of tenacious soil or solids. This is followed by immersion in cold solvent to cool the parts. Then follows exposure to condensation of hot, clean solvent vapors on the work. This final step also removes any last traces of oil and grease and dries the part. For removal of caked-on oils and compounds, a predip in cool solvent can be used to wet and loosen the soil before the degreasing operation.

10.3.1 Vapor degreasing can be used to clean all types of metal, including steel, steel alloys, light metal alloys, special bronze, nonferrous metals, nickel, and titanium. This method simplifies the cleaning of parts containing several metals because it cleans by solvent action instead of chemical action; there is no danger of over-cleaning or under-cleaning because of any difference in chemical activity of the metals present. Because of the rapid penetrating action of the solvent and solvent vapor, this method is effective in cleaning parts containing recesses, blind holes, perforations, crevices, and welded seams. Where the soils are present on surfaces that are not readily accessible, the process is sometimes supplemented by ultrasonic cleaning in the solvent rinse chamber.

10.3.2 Vapor degreasing is effective on solvent-soluble soils and chemically active lubricants. Insoluble soils (buffing grits,

metal chips and dust, etc.) are flushed away as the soluble soils (greases and oils) dissolve in the solvent. It is not effective on metallic salts, scale, carbon deposits, many inorganic soldering or welding fluxes, and fingerprints unaccompanied by oil or grease. This process is frequently competitive in cost with wet cleaning methods. Its lower equipment, floor space, and heat requirements offset the higher cost of solvent.

10.3.3 For some applications (steel stampings, buffed zincbase die castings, etc.), the degreased work can go directly to mild electrolytic cleaning and subsequent electroplating without the need for an intermediate alkaline cleaning step.

10.4 Emulsion Cleaners—Oils and high-boiling hydrocarbons such as kerosene have the ability to dissolve most greases, particularly at high temperatures. The addition of emulsifiers, soaps, and wetting agents enhances the penetrating power of the organic solvent and permits removal of the latter and associated soil by power flushing. Further, intimate contact of the metal surface with the aqueous phase permits removal of materials not soluble in the hydrocarbon phase.

10.4.1 The principle of emulsion cleaning can be applied in a variety of ways including the use of straight emulsifiable solvents, unstable emulsions (diphase cleaners), invert-type emulsion cleaners, and stable emulsions. Additions of rust inhibitors or of alkali cleaners can be made to the water phase. Since agitation is important to good cleaning, the power-spray cleaners find wide applications.

10.4.2 Emulsion cleaners are used at temperatures up to 82°C. The higher temperatures remove soils more quickly and effectively, but caution must be used with cleaners containing organics of low flash point. Some cleaners containing chlorinated solvents are used above the flash point of some of the components since the chlorinated portion will volatilize to extinguish flashes.

10.5 Biological Cleaners (8)—Highly emulsifying soak cleaners are combined with living microorganisms to permit the removed oils, greases, and other complex organic compounds to undergo a natural process known as bioremediation. Living microbes break down organic compounds, such as oil and grease into carbon dioxide and water and the cleaners, if properly maintained, may run for years without changing the bath at all. Since the cleaning fluid is kept free from contaminants, the such systems allow more effective cleaning for a greater length of time.

10.5.1 In order to maintain a healthy biosystem, operating conditions are critical. Typically, optimum pH range for these types of cleaning systems is 8.5 to 9.5. Too high a pH will result in lowering of the bacteria action, and oil will be built-up. Too low a pH will render the bacteria too active resulting in consumption of the wetter and other organics necessary for proper cleaning. Temperature also is a critical operating parameter. Optimum metabolism of oil and grease is achieved around 40 to 50°C.

10.5.2 Air agitation is critical to maintaining an oxygenated environment to maintain sufficient biological activity and only aerobic bacteria. Without air during operation, anerobic bacteria are produced and the cleaner will take on a noticeable, unpleasant odor. Air sparging also improves overall cleaning efficiency by promoting transfer of oil and grease particles

from part surface into the cleaning solution. In order for bioremediation to proceed, particles must be detached from the part surface.

11. Precautions

- 11.1 The use of solvents and emulsions of diphase cleaners requires special attention to safety hazards. Petroleum and aromatic solvents of low flash point, for example less the 55°C, must be used with caution. Underwriters Laboratories-approved containers and adequate ventilation should be provided to avoid the accumulation of fumes in explosive concentrations. Diluted emulsion cleaners usually have flash points above 70°C and emulsifiable solvents of high flash point are now available.
- 11.2 Trichloroethylene and perchloroethylene are nonflammable under the conditions of the vapor degreasing process and are among the least toxic of the chlorinated hydrocarbons; up to 100 ppm of either is tolerable in the working atmosphere for a normal 8-h working day. Trichlorotrifluroethane (1000 ppm tolerable limit) is also used. With proper equipment design and operation, solvent vapors in the working area are easily maintained well below recommended safe limits. Degreaser tanks should preferably be cleaned and maintained from outside the tank. Entry into a tank should be made only after all solvent and vapors have been removed and then *only with an observer* on the outside. Proper ventilation cannot be overstressed because workmen will often discard a recommended gas mask. For cold-solvent operations, adequate ventilation must be provided in the work area.
- 11.3 Because soils accumulate in solvents, the solvents must be discarded or purified by distillation. In vapor degreasing equipment, the solvent is recovered by distillation and the soil discarded. The use of automatic auxiliary stills in conjunction with the degreaser allows continuous cleaning operation and solvent recovery.
- 11.4 Emulsifiable solvents must be discarded occasionally, although frequently most of the soil is flushed off in the rinse. Emulsion cleaners represent a particular problem of bath contamination because of the lack of adequate analytical controls to determine bath life. Because emulsion cleaners yield a water-shedding surface, the effect on water-break due to accumulated oils is difficult to differentiate from that due to the solvent. Because of the low cost of diluted emulsion cleaners. it is economical to discard these baths at frequent intervals. Soap-base emulsion cleaners can cause difficulties where acidic soils are introduced; here mixed alkalies and emulsion cleaners can also require water conditioning in hard-water areas to avoid precipitation of hard-water soaps. Good housekeeping is desirable to avoid bacterial contamination of emulsion cleaners. Bacteriostats can be included in the formulations of cleaners to prevent the unpleasant odors that result from bacterial action.
- 11.5 As indicated in 14.7.8, disposal of emulsion cleaners can present problems.

PART II—INTERMEDIATE (ALKALINE) CLEANING

12. Purpose

- 12.1 Intermediate alkaline cleaning removes solvent residues and residual soil which has been softened or conditioned by precleaning. Spray or soak alkaline cleaning also can be used as a precleaning stage followed by additional alkaline cleaning, if the soil and metal lend themselves to this treatment. This is not so for metals that are sensitive to alkaline cleaning, such as zinc, because the time in the alkaline cleaner should be minimal. Some electroplaters use the term precleaning for alkaline cleaning before electrocleaning, especially when solvent cleaning is carried out at a different part of the plant.
- 12.2 Although industrial practice is limited, vapor degreasing alone is sometimes used before electrocleaning. Most oils and greases and some buffing and drawing compounds are effectively removed and contamination of the electrocleaning bath is lessened. The specific applications will not be given in detail here (5, 6, 7). Manufacturers of degreasing solvent or equipment should be consulted for details.

13. Types

- 13.1 Soak alkaline cleaning is carried out at 30 to 120 g/L of alkaline cleaner at temperatures of 82°C to boiling, for periods of 3 to 15 min. If used ultrasonically, temperatures may be 70°C to boiling. The cleaners usually contain surface-active, soap-like materials which foam if agitated vigorously.
- 13.2 Spray alkaline cleaning is usually carried out at 4.0 to 15 g/L at temperatures of 50 to 82°C for 1 to 3 min with spray pressures of 69 to 345 kPa (10 to 50 psi). Foaming may be a problem, unless the cleaner is properly designed.
- 13.2.1 Foam is also a major problem because of accumulation of soaps in the cleaner from the action of the alkali on some organic soils and drag-in of wetters from precleaners. For this reason, it frequently is desirable to use low concentrations of cleaner, for example, 4.0 g/L, and to discard the solution often, even though cleaning is adequate. For the same reason, it is sometimes necessary to operate at lower pressures even though higher pressures give better cleaning.
- 13.3 Barrel alkaline cleaning is usually carried out at 7.5 to 45 g/L. Temperatures are usually lower than for soak cleaning because of mechanical factors. Although agitation is better than in some cleaning, control is frequently not as good.

14. Factors Influencing Good Alkaline Cleaning

- 14.1 *Concentration*—The optimum concentration of the cleaning solution should be determined by actual tests because many factors are involved.
- 14.2 *Temperature*—Best results are obtained near the boiling point if other conditions permit. The high temperature reduces the viscosity of the soil. A rolling boil provides agitation. In some cases, cleaner formulation may be such as to make lower temperatures optimal.
- 14.3 *Time*—Alkaline cleaners, operating by the mechanism of lifting the oil film, require a reasonable time to permit the surface-active materials to act on the surface. This time is

shortened if agitation is vigorous, temperatures high, and concentration high. Age of solution and contamination retard cleaning.

14.4 Agitation:

14.4.1 Spray Cleaning (2)—As in emulsion cleaning, much of the effectiveness of spray cleaners in removal of solids is due to the mechanical action of the solution sprayed on the surface. Hence, every effort should be made to obtain efficient impingement at high pressure without pushing the work out of the spray area (or off the racks). Foaming after soap accumulation also limits spray pressure. The action of spray alkaline cleaners depends on detergency as well as mechanical action; the proper materials and conditions should be used. Spray nozzles should not be allowed to clog with solids and vestibules should be provided at each stage of the machine to avoid contamination by overspray. Agitation is very good in spray cleaning (if the sprays hit the solid surfaces directly).

14.4.2 *Soak Cleaning* (3)—Suggested methods of agitation in soak cleaning are as follows:

14.4.2.1 Complete withdrawal and reimmersion, especially at about half the total time allotted in the cleaner,

14.4.2.2 Pumping the solution over the work with proper intake and adequate pressure.

14.4.2.3 Rapid motion of the work in the cleaner,

14.4.2.4 Maintenance of a rolling boil,

14.4.2.5 Use of an air line or a mixer,

14.4.2.6 Ultrasonic agitation, and

14.4.2.7 Use of an overflow weir assists in separating unemulsified oils, grease, and other solids.

Note 3—Electrocleaning represents a special method of agitation and is considered in 15.4.

14.5 Age and Degree of Contamination—A long bath life is desirable, not only for economy but also to avoid the necessity for constant supervision. Alkaline cleaners vary in this respect; much depends on the amount of soil present on the work being cleaned. Care must be taken to avoid redeposition of the soil as parts exit from the cleaner. Cleaners should be discarded periodically because of accumulation of debris, etc. Surface oil should be skimmed frequently, preferably by overflowing into a properly designed skim trough. The cleaner should be buffered to have a high tolerance for acidic soils. Tolerance for soaps, formed by reaction with the soils, can be designed into the cleaner when necessary.

14.6 Rinsing (see Section 6)—Double rinses are desirable to reduce the concentration of cleaner in the rinse. A first hot rinse gives more efficient cleaner removal, while a second cold rinse reduces the tendency to rusting and tarnishing during transfer to the next stage. If rinsing is inadequate, the components of the cleaner must be selected to promote easy rinsing. Selection of a free-rinsing cleaner may require some sacrifice in cleaning properties. Rinse tanks should have adequate overflow rate, skimming troughs of good design, and proper positioning of the intake water line.

14.6.1 Agitation of work in the rinse tank is desirable. Water must also flow through hot rinses, although a reduced rate is often desired for economy. Spray rinsing is very effective, especially if coupled with soak rinsing by having a spray of

clean water hit the work as it exits from the rinse tank. Drying of the cleaner on the work before rinsing should be avoided by having fog nozzles after cleaning tanks, if necessary, or by decreasing transfer time or operating temperatures. With some cleaners, adequate rinsing is difficult to obtain after the cleaner has dried on the work.

14.7 Selection of the Cleaner—Important factors to be considered are as follows:

14.7.1 *Soil* (see 3.1 and 3.2)—If the soil is rich in soaps, the cleaner should be lean in this respect and should have built-in high soap tolerance. Because of the specific action of the individual cleaners on certain soils, this is a prime factor in cleaner selection.

14.7.2 *Metal* (see 4.1)—Special cleaners are designed to avoid harmful effects on zinc, aluminum, and brasses. Iron, steel, magnesium, and copper have better tolerance to alkaline cleaners.

14.7.3 *Water*—Hard water frequently requires cleaners rich in water softeners, such as sequestering and chelating agents, largely to prevent difficulties during rinsing.

14.7.4 Method of Application (4)—There is no universal cleaner that can be efficiently applied by all methods of cleaning. The cleaner should be formulated for its method of application. Cooperation of purchaser with vendor in permitting study of equipment, and availability of heating facilities will minimize problems such as incomplete cleaning, excessive foaming, dry down of cleaning solution before rinsing, etc.

14.7.5 Degree of Cleanliness Required (5)—The degree of cleanliness required for the next operation should be determined by the purchaser and explained to the vendor. If parts are to be held over between operations, a light oil or alkaline protective film may be advantageous to prevent rusting. Parts going immediately to electroplating should be free from any objectionable films that might prevent adhesion of electrodeposits.

14.7.6 *Unit Cost*—Many factors enter into the cost of a cleaning operation. Among them are: yearly cost of space occupied by the equipment, capital costs and amortization of equipment, utilities (water, heat, power, etc.), maintenance by laboratory control, make-up, additions, and disposal of spent solutions, labor, cost of cleaning compounds, and cost of reprocessing rejects. To obtain true costs per thousand square feet of work processed all these factors must be included.

14.7.7 *Safety*—Consideration must be given to the safety of operators and equipment and parts being cleaned (see 11.1, 11.2, 11.3, and Section 16).

14.7.8 Disposal of Spent Solutions and Rinse Waters—Local, state, and Federal regulations must be consulted before final selection of a cleaning material can be made. Increasing attention to reduction of stream pollution throughout the country means that this factor is a more decisive one in selection of cleaning materials. Examples of materials that come under such regulations are: chromates, cyanides, nonbiodegradable detergents, phenolics, spent solvent emulsions, and phosphates.

15.1 General—Actual compositions of cleaning materials for various metals, soils, methods of application, degree of cleanliness required, and other specific conditions are beyond the scope of this document. The fact that individual suppliers of proprietary cleaning materials have hundreds of standard products available indicates the complexity of this field. In like manner, the recommendations for their application vary widely. Some requirements for formulations of cleaning materials are listed below:

15.2 Soak Cleaners:

- 15.2.1 Ability to saponify animal and vegetable oils, if conditions such as paint stripping require it on steel parts. Otherwise, this requirement can be undesirable where sensitive metals are involved, because attack on the metal may occur thereby.
- 15.2.2 Wetting and emulsifying action supplied by soaps or synthetic surface agents to wet oily and greasy deposits and, with agitation, suspend the deposits in an emulsified state until rinsing is accomplished.
- 15.2.3 Deflocculating action or colloidal properties to attract solid particle soils and suspend them in a free rinsing condition, so that redeposition of the soil is prevented.
- 15.2.4 Water-softening ability to sequester or chelate calcium and magnesium ions by combining with them to form water soluble nonionic complexes.
- Note 4—Use of polyphosphate-type sequestering agents is limited to temperatures below 80° C, because decomposition into orthophosphates occurs above this temperature. Organic chelating agents are stable to above 100° C. Chelating agents also have the ability to form water-soluble complexes with di- and trivalent insoluble metallic salts, such as iron oxides. Thus, specially compounded soak cleaners can remove rust, heat scale, and iron or zinc phosphate deposits as well as oily or other soils.
- 15.2.5 Buffering action to maintain the optimum pH range, despite introduction of acidic or alkaline soils.
- 15.2.6 Proper inhibitors for prevention of attack of sensitive metals.
 - 15.3 Spray Cleaners:
- 15.3.1 The short duration of spray washing operations generally precludes necessity for inclusion of saponifying action.
- 15.3.2 Wetting and emulsifying action is incorporated in this type of cleaner, but to a much lesser degree than in soak cleaners, to prevent excessive foaming. Special low-foaming surface-active agents are generally used. Otherwise, antifoaming ingredients are a part of the formulations.
- 15.3.3 Deflocculating action, or use of colloidal materials, is beneficial in suspending solid particle soils. However, unless properly compounded, they can cause excessive foaming.
- 15.3.4 Water softening to prevent formation of insoluble calcium scales on heating coils, spray nozzles, etc., is a necessary requirement of these cleaners.
- 15.3.5 Because of the large area of work going through spray washing machines and the lower concentrations of cleaner used, buffering action is an essential part of a spray cleaner, to maintain effective life.
- 15.3.6 Depending on the type of metal parts being cleaned, an inhibitor may or may not be required.

- 15.4 Electrocleaners:
- 15.4.1 High electrical conductivity is essential. Sodium hydroxide alone could impart this property, but it lacks the detergent action to remove oils, greases, and solid particle soils. Therefore, other materials must be incorporated with the sodium hydroxide.
- 15.4.2 Wetting and emulsifying action supplied by nonionic (low-foaming) and some anionic surface-active agents are required to increase solubility of the nonionics. These must be in sufficient quantity to wet and emulsify residual oil and grease films remaining from precleaning operations. However, their concentration must be low enough to prevent excessive foaming caused by the evolution of hydrogen and oxygen at the electrodes. The type of foam produced should be "brittle," rather than tenacious, and short lived, but be able to prevent annoying spray from the alkaline solution from escaping into the atmosphere. The organic surface-active agents chosen should not be decomposed into tarry residues by heat, high alkalinity, or oxidation at anodes. They should have a minimum tendency to "deposit out" on the electrodes.
- 15.4.3 Deflocculating or colloidal properties are necessary to suspend solid particle soils removed from the work in a free-rinsing condition. Again, these agents should have a minimum tendency to "deposit out" on the electrodes.
- 15.4.4 Water-softening ability is required to prevent formation of insoluble soaps by combination of hard-water salts with soaps formed by reaction of soils and alkaline constituents. Such insoluble soaps adhering to surfaces decompose in subsequent acid dips and leave objectionable films of fatty acids on parts to be electroplated. The sequestering polyphosphates are used for this purpose where operating temperatures do not exceed their decomposition range, about 80°C. The organic chelating agents are stable and offer another advantage over the polyphosphates in that they solubilize light oxide films on the metal parts and provide a more active surface for subsequent electroplating.
- 15.4.5 Buffering action is required in electrocleaners as in other types to maintain the optimum pH range, despite introduction of acidic soils.
- 15.4.6 Inhibition to prevent attack of sensitive metals is required for the undesirable condition where a single electrocleaning solution is used as a precleaner and final cleaner. On ferrous alloys no inhibition is required. Where sensitive metals have been precleaned, a slight attack or oxidation of the surface, when anodically cleaned, is considered beneficial. This action, plus subsequent acid dip, before electroplating, removes disturbed metal surfaces and promotes better adhesion.

16. Precautions (9)

16.1 Some alkaline cleaners generate considerable heat when dissolved in water. Rate of solution, however, is frequently very slow in cold water. It is most advantageous to have the water at a temperature of about 50°C while the cleaner is carefully sprinkled in small quantities over the surface of the tank, as with a shovel. Others prefer the safer but slower process of adding the cleaner directly to the tank of water at room temperature with provision for agitation to avoid caking.

A caked cleaner dissolves slowly. If alkali gets on the skin, it should be washed profusely with water and medical attention received promptly.

PART III—FINAL ELECTROCLEANING

17. Purpose

- 17.1 The objectives of metal cleaning before electroplating have been summarized as follows (10): In electrodeposition a surface is required which will receive a smooth, adherent metal deposit, but this is not necessarily an absolutely clean surface. In general, an acceptable surface is one on which objectionable surface films have been replaced by films more suitable and acceptable for electroplating.
- 17.2 Assuming that the metal parts have been precleaned by methods described above, the "objectionable surface films" are precleaner residues, minor amounts of soils such as oils and solid particles not completely removed by previous cleaning treatments. Thus, the final electrocleaning process is insurance that only films remain that are" more suitable and acceptable" for the electroplating operations.

18. Types of Electrocleaning

- 18.1 Electrocleaning is soak cleaning with agitation provided by the upward movement of bubbles of hydrogen or oxygen formed by the electrolytic decomposition of water in the solution. Because of this electrolytic action special types of cleaners are required (see 15.4).
- 18.2 *Cathodic*—Parts negatively charged in the electrocleaner tank are cathodically or direct current cleaned. Hydrogen gas is evolved on the surface. Positively charged ions and colloids are attracted to the cathode.
- 18.2.1 Cathodic cleaning provides greater agitation be cause two volumes of hydrogen are evolved on the surface, as compared with one volume of oxygen at the anode. In cathodic cleaning, little or no tarnish or attach of nonferrous metal occurs.
- 18.2.2 Cathodic cleaning attracts positively charged metallic ions, soaps, and other colloidal materials in the solution causing them to deposit out as loose smuts on parts being cleaned. Since hydrogen is evolved on the parts it may penetrate and become occluded in hardened steel parts, causing embrittlement. Cathodic electrocleaning solutions are more sensitive to chromic acid contamination than anodic electrocleaning solutions.
- 18.3 *Anodic*—Parts positively charged in the electrocleaner solution are anodically or reverse current cleaned. Oxygen gas is evolved on the surface. Negatively charged ions and colloids are attracted to the parts.
- 18.3.1 A higher degree of cleanliness is obtained owing to the "deplating" action resulting from the positively charged surface repelling positively charged metallic ions, soils, and smuts. Oxygen gas does not enter metals as hydrogen does. Anodic electrocleaning solutions have much more tolerance for chromic acid contamination.
- 18.3.2 Less agitation occurs at the anode surface because only one volume of oxygen gas is evolved from the decomposition of the water.

- Note 5—Nonferrous metals are tarnished or attacked if anodic cleaning is prolonged in an *uninhibited* electrocleaning solution. However, this action may be beneficial as discussed in 15.4.6. Alloys of lead, nickel, or silver and nickel, and silver electroplated surfaces should not be anodically cleaned. Lead alloys are rapidly attacked and nickel and silver surfaces become passivated, requiring special activating treatments for subsequent electrodeposits.
- 18.4 Periodic Reverse (PR) Current Cleaning—A modification of normal electrocleaning methods is the use of a number of cycles of periodic reverse current to assist in removal of soils, instead of a single application of direct or reverse current cleaning, or a combination of them. Details of electrical equipment for production of periodic reverse current are outlined in a recent book (11). Users of PR cleaning, unlike PR electroplating, prefer a PR cycle in which the cathodic (directcurrent) time equals the anodic (reverse-current) time. To prevent deposition of loose metallic smuts, the work should be removed from the electrocleaner during the deanodic portion of a cycle. PR cleaning gives improved smut removal, accelerates cleaning operations, and provides a more active surface for subsequent electroplating (12). PR cleaning improves electrolytic removal of rust and scale in alkaline chelating solutions **(13)**.

PART IV—TROUBLE SHOOTING

19. General

- 19.1 Trouble in cleaning may be quite obvious or may be very obscure. Electroplating operations generally are complex and consist of many steps, so that very often it is not obvious whether trouble is due to cleaning or some other phase of the electroplating operation. It can be accurately stated, however, that many electroplating troubles can be traced to faulty cleaning. When trouble occurs and faulty cleaning is suspected, an initial close observation of the work should be made after each cleaning and rinsing step. Things to look for are water breaks (especially after acid dips), excessive darkening or etching, smuts, irregularity in appearance, and stains. Good lighting is required for accurate observation, and also, the observer should be one who knows from experience how correctly cleaned parts should look after each cleaning step. Parts emerging from rinses and acid dips should also be examined for evidence of films either being picked up or not being removed.
- 19.2 Cleaner concentrations and any special standards recommended by the supplier such as pH also should be determined, along with tank temperatures, the possibility of tanks being skipped if it is a hand line, the current density in electrocleaners, polarity, time in tanks, and transfer times. These all have a bearing on the proper functioning of cleaners. If any of these have been changed, they are to be considered a possible source of the trouble.
- 19.3 The nature of the soils being removed must also be considered. If buffing or drawing compounds or oils have changed, the new soils may be more difficult to clean. Sometimes a change in soils may require a change in cleaners or a change in the method of cleaning. Likewise, a substantial increase in the quantity of soil, such as buffing compound, could cause trouble.

19.4 One good way to establish whether cleaning is the cause of the trouble is to hand scrub a few parts with a bristle brush and pumice powder, rinse, and then place them directly in the electroplating solution without cleaning or acid dipping. If the same trouble is evident on these parts, it can usually be concluded that the trouble is in the electroplating baths or elsewhere and not in the preparatory part of the cycle.

20. Detecting Trouble in Specific Cleaning Operations

- 20.1 Soak or Hot Still-Tank Cleaning:
- 20.1.1 *Heating*—Check the temperature. There should be sufficient heating capacity to keep the solution at the proper temperature during full production. If steam coils are used, check for leaks diluting the solution. Check that the heating element is not insulated from the solution by scale or sludge. Check the operation of the thermostat.
- 20.1.2 Agitation Shield and Overflow Dam—The solution should be properly agitated by bringing fresh cleaner solution continuously to the work. The soils should be skimmed from the solution surface over the dam.
- 20.1.3 *Contamination*—Heavier production can cause undue soil loading of the cleaner. Check the age of the cleaner. Investigate introduction of any new soils that might cause the cleaner to become exhausted prematurely.
 - 20.2 Spray Washing Machines:
- 20.2.1 *Heating*—Check thermostats, scale on heating coils or tubes, and solution temperature. Descale the machine, if necessary, with inhibited hydrochloric acid or a proprietary descaling chemical to improve heating efficiency.
- 20.2.2 *Spray Risers*—There should be enough spray risers, properly positioned and directed to contact all surfaces. The nozzles should be periodically cleaned.
- 20.2.3 *Pumps and Lines*—Check on the return side to be sure no air is being sucked into the system causing excessive foaming.
- 20.2.4 *Baffles*—The cleaning and rinsing stages must be properly baffled to prevent one from contaminating or diluting the other.
- 20.2.5 *Contamination*—Excessively dirty cleaning solutions cannot be expected to function properly. Grease overflow dams must function properly. Production must not be excessive before a new cleaner is made up.

20.3 Electrocleaners:

20.3.1 *Current Density*—Too high a current density when cleaning metals anodically may etch a buffed surface or cause excessive tarnishing. Too high a current density when electrocleaning cathodically will cause electroplating out of dissolved metals and some colloids as a smut. Too low a current density (cathodic or anodic) can be the cause of poor cleaning. Check the current with tong ammeters if available and calculate current density. Good steel electrocleaning requires a minimum of 5 A/dm²; 8 to 10 is better. Brass and zinc die castings and nickel deposits normally are cleaned at 2 to 4 A/dm².

- 20.3.2 *Bus Bars*—Be sure that the bus bars are of ample size to carry the current required for a work load (normally 158 A/cm². cross section of copper bar). Be sure that the bus bars are clean to ensure good contact.
- 20.3.3 *Polarity*—Be sure that the polarity is proper for the job. Cathodic cleaning should seldom be used as a final electrocleaning step because of the danger of "depositing out" dissolved metals. There are exceptions to this rule, as in electrocleaning lead, magnesium, nickel, silver, and some stainless steels. If doubt exists as to polarity, check with a voltmeter, or trace back the conductors to the current source.
- 20.3.4 *Insulation*—Be sure that the tank is insulated from the floor, and that the work, electrode rods, and electrodes are insulated from the tank. Insulated joints on steam, drain, and water lines should be provided.
- 20.4 *Rinses*—Rinses should be kept clean and overflowing. Some cleaners require warm rinses for maximum efficiency. The cleaner supplier's instructions should be followed. It is poor practice to use a common rinse tank for several purposes, such as following a soak cleaner, electrocleaner, and an acid dip. Air agitation in rinses is beneficial.

21. Failures Attributable to Cleaning

Note 6—There are a number of other reasons for these failures, not associated with cleaning.

- 21.1 Blisters, Peeling, or Poor Adhesion—These defects may be caused by improper polarity in the electrocleaner, overcleaning (current density too high or cleaning time too long), no current in the electrocleaner, and oil and grease not completely removed. In certain circumstances, especially with materials used in the electronic industry, such electroplating defects may arise from other factors as well.
- 21.2 *Pitting*—Pitting may be due to oil and grease not being completely removed. If pitting is general and on all parts, the trouble is probably in the electroplating tanks.
- 21.3 Strains—This is a problem associated with bright-electroplated work. The most common cause is cleaner drying on the work during transfer. It can be eliminated by lowering the cleaner temperature, decreasing transfer time, installing fog nozzles to keep parts wet until they reach rinse tanks, or by using cleaners designed to prevent staining of sensitive non-ferrous metals. It also can be caused by incomplete soil removal, particularly tightly adherent oils of a polar type almost impossible to remove in alkaline cleaners. Solvent degreasing or emulsifiable solvent predips may be required.
- 21.4 Roughness—This is caused by failure to remove smut or other solid particles. Sometimes this can be traced back to vapor degreasing which removes the oil but not the solid particles. If this is a cause, a high-pressure solvent spray will often help. Roughness also may be caused by incomplete rinsing of alkaline soak or electrocleaners due to excessive water hardness or an exhausted cleaner bath. Magnetically charged particles can cause roughness on electroplated steel parts.

REFERENCES

- (1) Kushner, J. B., *Plating*, Vol 38, 1951, pp. 933–935.
- (2) Lux, G. A., and Linford, H. B., Electroplating Engineering Handbook, Graham, A. K., Editor, 2nd ed., Reinhold Publishing Co., New York, NY, 1962, pp. 153–176.
- (3) Spring, S., Metal Cleaning, Reinhold Publishing Co., New York, NY, 1963.
- (4) Linford, H. B., and Saubestre, E. B., Plating, Vol 37, 1950, p. 1265: Linford, H. B., and Saubestre, E. B., in AES Research Report, Serial No. 18, "Cleaning and Preparation of Metals for Electroplating," American Electroplaters' Society, Newark, NJ.
- (5) Baker, M. E., and Hetrick, G. H., Electroplating Engineering Handbook, Graham, A. K., Editor, 2nd ed., Reinhold Publishing Co., 1962, pp. 126–153.
- (6) ASTM Committee D26 Cold Cleaning with Halogenated Solvents, ASTM STP 403, ASTM, Philadelphia, PA 19103.
- (7) ASTM Committee D26 Handbook of Vapor Degreasing, ASTM STP

- 310, ASTM, Philadelphia, PA 19103.
- (8) McNally, T. W., Parts Cleaning, Witter Publishing, Flemington, NJ, p. 20–27.
- (9) Spring, S., Metal Cleaning, Reinhold Publishing Co., New York, NY, 1963, pp. 202–207.
- (10) Lyons, E. H., *Transactions*, Electrochemical Society, Vol 88, 1945, p. 281.
- (11) Ceresa, M., Electroplating Engineering Handbook, Graham, A. K., Editor, 2nd ed., Reinhold Publishing Co., New York, NY, 1962, pp. 689–704.
- (12) Spring, S., *Metal Cleaning*, Reinhold Publishing Co., New York, NY, 1963, p. 140.
- (13) U.S. Patent 2,915,444, Dec. 1, 1959.
- (14) "Heat Treating, Cleaning and Finishing," *Metals Handbook*, Vol 2, 8th ed., pp. 320, 616, and 640.

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