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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—Pretreatment (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).

3.1.4 Machining oils.

3.1.5 Rust-preventive slushing oils or greases.

3.1.6 Electroplater's stop-off residues.

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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 pre-cleaning 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 adhere 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. Lower-viscosity 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 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.)

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

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 zinc-base 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 (14)*—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 – 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, anaerobic 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 diphasic cleaners requires special attention to safety hazards. Petroleum and aromatic solvents of low flash point, for example less than 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. Trichlorotrifluoroethane (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 alkalis 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