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Cleaning in the Metal-Processing Industry

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formidable task. Still greater difficulties arise in

attempting to devise meth-

ods of laboratory evalua-

tion capable of predicting

ing materials employed in

the metal-finishing industry is no exception and is

further subject to compli-

cations which are somewhat specific to that field.

The soils involved have

often resulted from prior metal-processing. With

proprietary products, such

as drawing compounds,

rust-inhibiting oils, etc., a

The evaluation of clean-

field results.

HE BASIC OBJECTIVE of every cleaning operation is to remove soil efficiently while not exceeding tolerable limits of attack upon the substrate. Translation of this simple statement into a list of practical requirements adequate either to define efficiency of soil removal or limits of attack can be a

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variety of soil conditions can exist among plants performing the same metal-working operation. Another distinction lies in the fact that eleaning procedures in this industry are followed by subsequent finishing operations which differ widely in their requirements for surface cleanliness and in which constituents of the cleaning materials can themselves serve as soils.

Some idea of the scope of metal finishing and its cleaning requirements can be gained from an examination of Table I in which the principal variables are listed. Not included are certain metals now produced in small quantities, which, in the following years, may become as important as some shown here. Ultrasonic cleaning, which would not have appeared a few years ago, is now assuming greater importance. The list cannot therefore be regarded as complete and unchanging and serves only as a partial example of current practice.

The metal surfaces, important from the standpoint of limiting the cleaning compositions which can be employed without excessive corrosion, are equally important in the sense that they can become an integral part of the soil either in finely divided metallic form or by reaction with the other soil constituents. The soils derived from a given metal-working operation, such as drawing, can vary from simple soap films removable by hot water to coatings containing pigments, oils, and additives which cling to the metal surface during severe forming operations and often, unfortunately, during the subsequent cleaning procedures. Since it is common to perform more than one operation in the course of fabricating parts, mixtures of soils frequently occur.

The manner in which cleaning compounds are employed differ primarily in their use of detergent, solvent, chemical, electro-chemical, and mechanical effects, whichever is more effective. Economic aspects, weighed against the time available for cleaning, and the degree of ultimate cleanliness required are also involved.

At the same time the manner of use places requirements upon the cleaning material not directly related to cleaning ability but no less important from the standpoint of proper performance. Plating generally requires the highest degree of surface cleanliness, but even here some types of solutions show a far greater sensitivity toward soil than others. Other finishing operations, such as porcelain enamelling, may be as sensitive as plating to certain types of soil and yet be wholly insensitive to others.

Viewed broadly, therefore, the evaluation of cleaning materials in the metal-finishing industry can be exceedingly complex. Properly speaking, their properties can be defined only with reference to the particular operation in question. In the multiplicity of requirements however, certain general properties recur, some of the most important of which follow: soil removal, retention of cleaning ability in continued use, effect of constituents of the cleaner upon succeeding operations, control of foaming, and corrosion.

The above do not include many properties of importance to metal-cleaning materials, such as toxicity, flammability, effects of water hardness, emulsion stability, etc. They do however represent areas in which evaluation procedures can be most difficult and, if conducted improperly, quite misleading. The following discussion will therefore be limited to the laboratory evaluation of the factors shown with reference to practical plant conditions. A comprehensive review of metal cleaning is contained in an excellent series of bibliographical abstracts prepared by J. C. Harris (5) .

Soil Removal

An examination of the soil removal properties of a eleaner must satisfy the following requirements: the soil or soils must be representative of those to be re-

TABLE I Variahles Involved in Metal-Cleaning Operatiens

Substrates	Soils derived from metal-working processes	Manner of cleaner use	Subsequent operations
	Drawing and forming Drawing compounds Extrusion Carbon from process Machining Cutting oils Polishing Abrasive grit, grease stick Buffing Buffing compounds Lapping and grinding Grinding lubricant, soluble oils, abrasive Barrel finishing Metallic soaps Painting Rejects, paint over-spray Casting Mold-release compounds Mill operations Preservative oils, mill markings, conversion coatings Heat treating Quenching oils Scale	Hand wiping Still tank Spray washing Tumble washing Electrocleaning Ultrasonic cleaning Vapor degreasing	Plating Copper Nickel Chromium Zinc Cadmium Painting Conversion coating Paint application Porcelain enamelling Chemical milling Anodizing Welding

moved in practice; the soil must be applied to the proper metal substrate in such a manner as to yield a representatively soiled surface; the cleaning procedure itself must include all significant variables found in plant practice; and the interpretation of cleaning efficiency should be made in terms of the effects o'f soil residues upon the succeeding operation.

Where a cleaning material is tested in a plant process, all of these conditions are automatically fulfilled. Parts flowing from the production line through the cleaning process represent the first three requirements, provided a sufficient volume of parts are processed to represent normal variation in plant operations. The fourth requirement is satisfied by an examination of the work after the finishing operation. Unfortunately, should the results at that point prove unsatisfactory, the test can become a rather expensive experiment. A close approximation of plant practice can be achieved by processing parts in a pilot line, thus confining the test to a small but fairly representative portion of plant production. This is often impossible either because of the unavailability of equipment or because the size of the parts precludes such a test. In most cases there is little alternative but to attempt to establish an arbitrary laboratory test which will indicate, upon a comparative basis at least, the probability of successful plant operation.

Laboratory specimens are selected for this purpose from representative small parts or prepared by cutting panels from sheet stock to a convenient size for beaker tests. These are cleaned by the use of solvents or by other means prior to the application of processing materials taken from the plant. If the test soil is fluid, the panels can be coated by immersing them in the soil and withdrawing them at a fixed rate by such equipment as the Fisher Payne Dip Coater (1) or simply by dipping them and allowing them to drain for an arbitrary period. This procedure can also be used with more viscous materials' by thinning them with a suitable solvent diluent.

Still another method of preparing uniformly soiled panels, one frequently used in our laboratories, employs a gelatin printer's roller by which soil applied to a master panel is transferred to the laboratory panels. Occasional determination of the soil weight present on the specimens will show whether greater or lesser quantities should be applied to the master panel to compensate for that transferred to the test specimens. This technique is illustrated in Figure 1. With a small amount of practice this procedure is well adapted to the preparation of a large quantity of panels for routine tests.

Where the soil consists of buffing or polishing compounds, the manner of application can become considerably more involved. In this case the soil present on plant parts bears little resemblance to the initial composition of the buffing compound because of the reaction of the fatty acid binder with finely divided metal abraded from the substrate. There is therefore no alternative but to prepare these specimens by using a buffing machine of adequate size and apply specimens with the proper degree and duration of pressure to yield a representative soil condition. At best, it is difficult to obtain reproducible panels by this method, and it is usually necessary to prepare a large number and to divide these into groups, each showing approximately the same gradation of soiling. It should be noted also that buffing compound carried by the wheel to unbuffed surfaces, over edges, or impacted in crevices is considerably more difficult to remove than that present upon the highly buffed surfaces.

The age of the soiled panels can greatly affect the time required for the cleaning operation. This appears attributable in some cases to simple loss of solvent or water, air oxidation, or to a slow but continuing reaction with the substrate. This phenomenon can be as much a plant as a laboratoy problem, and it has been frequently noted that parts cleaned immediately after processing represent a less difficult problem than those which have been stored for some time prior to cleaning. Where this is true, preparation of laboratory panels a few days prior to performing the laboratory cleaning tests can result in a better correlation with actual plant conditions.

Having established a reproducible method of applying a representative soil to a reasonably representative surface, the method of conducting the cleaning test itself assumes equal importance. Simple cleaning operations such as those conducted in soak tanks are most easily correlated; the major difference between the laboratory and plant results is found in the time required for complete soit removal. These differences will be caused in large part by the presence or absence of mechanical dislodgement arising from circulation of the solution by convection currents from heating coils or restriction of that circulation by close packing of the parts in baskets. A laboratory comparison of a product having known cleaning properties in the plant with the one under investigation will generally offer a reliable prediction of the plant behavior of the latter.

The more complex the cleaning procedure becomes, the more seriously the results can deviate from those to be expected in plant operation. An example of this may be found in the laboratory evaluation of electrocleaners. Cleaning in this case is facilitated by mechanical and electrochemical effects produced by passage of an electric current through the part, the

FIG. 1. Roller method of soiling panels.

solution, and the tank electrodes. While it is common to measure one condition of this process by defining the current density in terms of the toal amperes of current flow divided by the surface area of the part, the actual current density in recesses may be so low as to be negligible while that present upon projections may be many times as great as the arithmetical average. Since reliance is being placed upon the flow of current to assist cleaning, it follows that this will not proceed equally well on all portions of the surface, and cleaning in recesses may more nearly approximate that of simple soak action. If tests are made with flat laboratory panels where some semblance of the calculated current density actually is present, these should include a wide range of current densities rather than the average figure indicated by the plant operation.

A similar problem exists in attempting to duplicate spray-washer operations by laboratory procedures. Since this operation depends upon the impact of the cleaning solution in assisting to dislodge the soil, the design of the nozzle is equally as important as the pressure exerted by the pump at that point. If the solution is largely atomized, the mechanical effect will be far less than if an equivalent volume of solution were projected more nearly as a solid stream. Another difficulty lies in the fact that plant parts may be such a shape that portions of the surface do not receive the full impact of the spray and are subject to little more than a flush with the cleaning solution. Attempts to correlate plant practice with tests made upon flat, laboratory prepared panels in such eases can be quite misleading. While some compensation can be made by using laboratory spray impact less than that indicated by plant conditions, considerable judgment is required in interpreting the results. Wherever possible, it is preferable to make the tests upon the actual parts.

Upon completion of the laboratory cleaning operations, a simple examination for gross residues may be adequate in some instances. For example, failure to remove buffing compounds from crevices prior to the electrocleaning operation virtually insures that the bulk of these will proceed on into the plating tanks.

Where the soils are naturally fluorescent, the use of ultraviolet light as described by Morgan and Lankler (9) affords a convenient and sensitive means of comparing the effectiveness of cleaning solutions and has the asset of showing not only the approximate quantity of soil residue but its distribution. This test is particularly well adapted for evaluating the results of soak cleaning operations. Where the soils are not naturally fluorescent, they can be made so by the addition of fluorescent dyes. This procedure carries with it the hazard that the dye will be extracted selectively by the cleaning process, and some experimentation may be necessary to establish that this does not occur before a suitable dye can be selected.

The most time-honored of all cleaning evaluation techniques is the use of the water break test, where it is assumed that a surface completely wetted by water is free at least of hydrophobic soils, such as oils or fatty acids. The atomizer water break test developed by Linford and Saubestre (7) yields a numerical rating of cleaning effectiveness in terms of the percentage of panel area completely free of water breaks. Alternatively, and as more commonly practiced, it is required that the surface be cleaned within a given time or set of conditions to the degree that water breaks are entirely absent. The latter is the most common criterion for cleanliness in metal-cleaning operations.

The radioactive tracer method described by Hensley and Ring (6) affords the most sensitive measurement of soil residues. The test restricts somewhat the types of soil systems which can be investigated and is presently most useful in fundamental cleaning studies. While radioactive soil constituents, such as stearic acid or hydrocarbons, can be incorporated into plant soils, the final evaluation will indicate only the amount of the tagged soil residue and not necessarily the total quantity of soil remaining on the surface.

The techniques described above are mainly directed to the detection of residual levels of hydrophobie soil. The presence of finely divided particulate matter, commonly called "smut," can be equally important where plating operations follow. The latter consists essentially of abrasives and finely divided metal arising from polishing operations. Unless removed by the cleaning operation or the acid treatment which follows, the subsequently applied plate will be cloudy or dull. Evaluation usually consists of wiping the cleaned surface with white cloth or absorbent tissue to indicate the presence and degree of this material left on the surface. A still better method is actually to plate the cleaned specimens.

While some of the evaluation techniques described above are highly sensitive, there is a dearth of information concerning the actual soil level which can be tolerated by some o'f the succeeding finishing operations. The absence of a water break probably represents a reasonably valid condition for cleanliness in some industries, such as porcelain enamelling, where the frit constituents are applied as a water suspension. Unless the latter can wet the prepared surface uniformly, it will either not cover these areas or may bridge only to spall off in succeeding drying and firing operations. The degree of cleanliness required for other finishing processes, such as plating, is less well resolved.

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Some time ago in our laboratory experiments were performed to' measure the effects of various soil residues left on metal surfaces upon the adhesion and appearance of a plate applied from a Watt's nickel bath. The results of these tests appear in Table II. These

data show that the plating bath has greater sensitivity for the more polar soils, such as the fatty acids, than for nonpolar substances, such as mineral oil. From this it may be inferred that the type of soil residue is no less important than its quantity.

A further complication exists in the fact that small quantities of soil present on the parts, while not in themselves sufficinetly harmful to produce apparent failures, may ultimately contaminate the plating bath itself to a sufficient degree to interfere with its proper operation. Table III illustrates the effect of adding various substances to two proprietary bright copper baths. Here the effects are specific for the type of plating solution as well as for the soil contaminent.

In the work done by Hensley and Ring, utilizing radioactive tracer techniques, an effort was made to determine the adverse effect of the soil residues upon adhesion of subsequently applied electroplates. Surprisingly good adhesion was obtained in many cases despite the presence of significant levels of stearic acid residues. At least part of the answer to this apparent contradiction will be found in an examination of Figure 2: a radio-autograph of a metal panel, where it can be seen that the residual soil is largely concentrated in the scratches on the metal surface and that a rather large percentage of the total area has been cleaned. Since our present techniques for measuring the adhesion of electroplates discriminate well only between poor bonds and good bonds and are not too sensitive to differences between good and excellent plate adhesion, the adverse effects of the soil in this case would not be too apparent.

FIG. 2. Radio-autograph of partially cleaned specimen.

Retention of Cleaning Ability in Continued Use

Since most cleaning solutions employed in the metalfinishing industry are subject to re-use for considerable lengths of time, the retention of cleaning ability is no less important than initial capabilities. This property can be measured in the manner described in the previous section either by repeating the cleaning tests with a large number of soiled panels or by the introduction of quantities of the soil itself. In either case the loss in cleaning effectiveness from this source can be determined by employing the same evaluation techniques. In plant practice however other effects also Occur which influence the usable life of the cleaning solution and cannot be measured directly by this test.

The degree of soil contamination of the cleaning solution can reach a level where, when carried into the rinsing operation, re-deposition will occur upon the cleaned surface. This effect can be influenced by reaction of rinse-water hardness with soaps employed in the cleaning products or those formed from soil constituents. In other cases, rinsing simply dilutes the contaminated cleaning solution to the point where it can no longer disperse the soil contamination. Small quantities of some soils carried still further down the line into the acid solutions normally used after the rinse will cause further damage. While the rinsing operation can be carefully controlled in laboratory tests, field operations are subject to wide variations in water hardness and, more importantly, in the degree of contamination maintained by still or overflowing rinse tanks.

In still other instances the failure of a cleaning solution is attributable in part at least to inadequacies of the control procedures employed to maintain it at operating strength. Since a considerable portion of the tank volume can be withdrawn by the passage of parts through it, quantities of water must be added to maintain the working level and appropriate quantities of Cleaning product added to maintain solution strength. While pH measurements may represent valid means of control for mildly acidic or mildly

alkaline cleaners, they have little, if any, value in the control of strongIy alkaline products where a pH greater than 12 can be maintained ranging from a concentration so low as to be inoperable to the limits of solubility. In such cases titration methods are most commonly used, and the choice of the end-point is dependent at least in part upon the purpose for which the cleaner is intended. Where high conductivity is required, as is true of electroeleaners for steel, titration to' the phenotphthalein end-point carries a certain degree of assurance that at least one of the most desirable properties of the solution is being' measured. In other instances where cleaning is not necessarily dependent solely upon high alkalinity, titration to the methyl orange end-point as a measurement of total alkalinity may have greater validity. At other times a combination of both measurements used simultaneously may offer still further information as to the probable condition of the cleaning solution. In the case of mildly alkaline materials, insistence upon maintaining a titration to the phenolphthalein end-point can result in a gradual increase in total solids content of the solution to the point that the effectiveness of the surfaetant content can be sharply reduced.

As a general statement, so many long-term effects can be involved in determining the life of a cleaning solution that laboratory evaluation offers at best incomplete information. In nearly every instance this must be determined on a specific plant basis.

Freedom from Constituents Which Can Adversely Affect Succeeding Operations

In certain instances the cleaning material itself can have an adverse effect upon the succeeding operation, either by undesirable reactions with the substrate or by carry-through of some constituents into the following operations.

It is common to apply a conversion coating to steel surfaces prior to painting; the zinc phosphates are one common type. The use of strongly alkaline cleaners to remove the soil prior to this operation can, while leaving very clean surfaces, yield coarse or powdery conversion coatings. The use of emulsion cleaners or very mild alkaline cleaners can promote the formation of better quality coatings. The cleaning of aluminum surfaces with silicate-inhibited cleaners prior to caustic etching can leave a film on the aluminmn part such that etching **is** delayed or proceeds in an uneven manner. In both cases it can properly be said that the cleaning operation itself is contributing to failure of the subsequent portion of the process.

The above effects are generally immediately and unhappily apparent. Less apparent, and equally disastrous long-term effects occur when certain constituents of the cleaner are carried down the processing line. The use of solvent cleaners to remove buffing compound residues has occasionally resulted in substantial quantities of the solvent being carried into the various plating solutions. Although good plant practice normally requires that solutions be maintained under conditions of constant filtration, this is not always the case, and sufficiently high levels of contamination will require that bath treatment be made. Simple examination of the cleaned parts for freedom from oil residues is not adequate since the racks from which the parts are suspended can carry significant quantities of this contamination. Since the latter are covered with an insulating rack coating, they are not subject to the final scavenging action of the electrocleaning' solution. The geometry of the part is also important since cup-like depressions or recesses can retain considerable quantities of the oil-containing solution and are poorly subjected to cleaning action by virtue of their shape.

The surfactant employed in an eleetrocleaner is subject to the same consideration since it is inevitable that small quantities will be carried into the acid solution, following the electroeleaner and the plating baths. Instability in acid solutions or incompatibility with plating-bath additives can cause poor plate adhesion or pitting.

The above situations represent only a few examples of the futility of attempting to divorce the cleaning procedure from succeeding operations. In all of these cases an examination of the cleaned surfaces could indicate complete success. Yet the results could be highly unsatisfactory from an operation standpoint.

Corrosion

The corrosion requirements for cleaning materials in the metal-finishing industries vary between limits where corrosion is to be entirely avoided insofar as possible to the point where corrosion, in one sense, is an integral part of the operation itself. For this reason attempts to measure or define limits of tolerable attack cannot be divorced from the specific application, and, as in the measurement of cleaning ability, all of the variables involved in the cleaning process must be considered. Since the susceptibility of various metals to attack by alkaline and acid environments is rather well known, this discussion will be restricted to a consideration of the effect of operating variables and field conditions upon metals subject to the action of conventional cleaning materials.

Simple immersion cleaning would appear to offer the fewest number of complications involved in determining the degree of corrosion resulting from contact with various cleaning solutions. For example, aluminum parts are commonly cleaned by immersion in mildly alkaline solutions inhibited by the use of sodium silicate. One A.S.T.M. method (2) sets forth a detailed procedure to be followed to determine corrosion in such cases. The corrosion results are noted in terms of weight change or alterations in the appearance of the surface of the speciment. A military specification (8) employs a measurment of the quanity of gas evolved by contact of the cleaning solution with a carefully prepared aluminum specimen as an indication of degree of attack. The latter test may not be accurate where the solutions contain oxidizing agents since consumption of the hydrogen gas under such conditions may indicate little corrosion despite the fact that attack has proceeded with vigor. Although tests conducted on laboratory specimens may show an inhibited alkaline cleaner to be completely free of corrosive action, plant parts ocasionally cleaned in such solutions may show severe localized etching accompanied by the formation of voluminous adherent white deposits in that area. Such attack can occur when the part has been stored in an environment causing slight and hardly visible corrosion. This effect can be duplicated with laboratory specimens by exposing them to conditions of high humidity and elevated temperature prior to the laboratory corrosion test. Specimens prepared in this manner will sustain etching in varying degree when cleaned in nearly all commercial silicate-inhibited aluminum cleaners.

Since the degree of attack can vary considerably, depending upon the severity of the corroding environment prior to the test, it is essential that plant parts employed as a basis for laboratory evaluation be taken from the same length of extrusion or from the same sheet of plant stock and that a sufficient number of specimens be treated in comparison solutions to offer a statistical basis for evaluation. The effect of process variables in more eomplex operation can be illustrated by the treatment of zinc die castings in electrocleaning solutions. A form of corrosion manifested by pitting or the formation of white deposits can occur with anodie electrolysis at low cleaner concentrations while high concentrations can produce excessive darkening or dulling of the cleaned surface. Sinee, as discussed under the measurment of cleaning ability, the current density distribution on a complex shaped object can range from nil to many times that indicated by the average current density, it follows that a complete laboratory examination of the corrosive behavior of a zinc electrocleaner should include a wide range of current densities within the concentration limits to be expected in plant practice. Another form of corrosion can occur during transfer of parts from the cleaning solution to the rinsing operation, where the cleaning solution dries upon the surface. In this drying procedure the concentration of the cleaner can increase many-fold above that of the cleaning solution itself. Relatively heavy sections may retain sufficient heat to cause both a high rate of attack and rapid concentration of the cleaning solution whereas thinner sections may cool more rapidly, reducing both the rate of attack and the degree of drying. The effect can be overlooked or minimized in tests employing small metal specimens or where the parts are transferred rapidly from the cleaning solution to the rinse water.

Control of Foaming

The degree of foaming which can be tolerated in a cleaning solution subject to varying degrees of agitation in a plant process can also be a limiting performance factor. Simple immersion cleaning procedures offer few problems in this respect unless air agitation is employed to facilitate soil dislodgement. Processes employing a high degree of agitation of the cleaning solution, such as electrocleaning or spray-washer operation, however, can impose severe restrictions upon both the type and quantity of surfactant which can be employed in the cleaning product. While an A.S.T.M. procedure (3) for determining the foaming characteristics of surfaetants is available, this does not afford much assistance in measuring the foaming properties of a produet in the latter two applications.

Where cleaning products are employed as eleetrocleaners, the evolution of hydrogen and oxygen from the work and the electrodes ear result in a copious aeeumulation of foam. When sparks are formed by breaking eontaet of racks, this foam can explode. While this is not ordinarily dangerous, it can be highly unnerving to personnel working near the tank. The total absence of foam can be equally undesirable since the operation may then produce an irritating alkaline spray. Surfaetants must therefore be carefully chosen to produce a foam adequate to prevent

fuming and yet breaking rapidly enough to prevent it from reaching the work rods. This property can be determined on a laboratory basis by simulating the eleetroeleaning operation in a manner described in Federal Specification P-C-535 (4). In performing such tests it must be remembered however that the degree of gas evolution and the consequent volume of the foam will be determined by the current density at the work and electrode and to some extent by the dimensions of the vessel in which the test is conducted. False indications of excessive foaming can be obtained by the use of unrealistically high current densities or by restricting the surface area to such a degree that the foam produced by the gas evolution cannot be dissipated by flowing away from the work, cooling, and breaking as it would in normal plant practice.

Foaming problems of still greater severity can be encountered in spray-washer operations. These in turn are dependent upon the degree to which the solution is agitated, which unfortunately, from a standard procedural standpoint, is dependent upon the washer construction. Products employed for this purpose commonly contain nonionie surfaetants as a means of limiting foaming while retaining detergent qualities. Since the solubility of these surfactants and hence their foaming characteristies are reduced both by increase in temperature and increase in the concentration of dissolved solids, the foaming properties should be determined with respect to the minimum operating temperature over the anticipated eoneentration range. A complication also resides in the fact that soils introduced into the spray washer can either promote or reduce the foaming of the solution. Performanee tests should therfore include the addition of plant soils at a reasonable level of contamination.

Summary

An attempt has been made to show the necessity of considering the cleaning procedures used in this industry in their relation to the entire process. It is a field unfortunately, where soil conditions, requirements for cleanliness, and many desirable operating characteristics are highly specific not only for a given process but for a given plant.

While artificial or reference soils are not entirely impractical in individual eases, they should be used with care and only where sufficient data exists to establish a working correlation with plant soils. Cleaning procedures themselves should be sufficiently broad in scope to encompass practical plant-operating ranges of time, temperature, concentration, and other significant variables rather than to rely upon "average" eonditions. The interpretation of the results of a cleaning operation should be made in the light of experience with the requirements of the succeeding operation, where, in some eases at least, a "clean" surface may not represent an adequate definition of acceptability.

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