

Specifying STAINLESS STEEL Surface Treatments

Though stainless steel is naturally passivated by exposure to air and other oxidizers, additional surface treatments often are needed to prevent corrosion.

Arthur H. Tuthill*,
Blacksburg, Va.,
Richard E. Avery*,
Londonderry, N.H.,
Consultants to Nickel Development Institute

Passivation, pickling, electropolishing, and in some circumstances, mechanical cleaning, are important surface treatments for the successful performance of stainless steel used for piping, pressure vessels, tanks, and machined parts in a wide variety of applications. Among them: pulp mills, nuclear power plants, hospital sterilization systems, food processing equipment, biotechnology processing plants, breweries, electronic-chip washing facilities, swimming-pool hardware, water treatment plants, and chemical process plants.

Determining which treatment should be used for specific applications is confusing to many specifiers. A good place to start is with ASTM A 380-88, "Cleaning and Descaling Stainless Steel Parts, Equipment and Systems,"¹ an excellent resource document for the cleaning of stainless steel, although it does not cover electropolishing.

Passivation treatments

Exposure to air is the natural, primary passivation treatment for stainless steel. This exposure produces a thin, durable chromium oxide film that forms rapidly on the alloy surface and gives stainless steel its characteristic "stainless" quality. Exposure of the surface to water or other oxidizing environments also produces this passivating film.

Additional passivation is called for in many specifications to remove light surface contamination from machined stainless steel parts, includ-

*Member of ASM International

ing shop dirt, iron particles from cutting tools, and machining lubricants. Passivation treatments of stainless steel with nitric or mild organic acids are useful mild cleaning operations performed after machining to enhance the protective nature of the natural, air-formed film. Nitric acid treatment enhances the level of chromium in the protective film on stainless steels.² DeBold has published an excellent practical review of nitric acid passivation of stainless steel machined parts.³

ASTM A 380 describes eight nitric-acid-based cleaning/passivation treatments and four cleaning treatments using other chemicals. None of these passivation treatments corrode or etch the surface. Several are designed to clean bright or polished surfaces by removing loosely adherent foreign matter. The most common treatment is

Handle with care

Most surface defects on stainless steel that are difficult to remove and, thus, contribute to corrosion, are produced during fabrication. Common defects include embedded steel particles, heat tint, arc strikes, weld spatter, grind marks, scratches, and organic contamination from marking crayons and paint. All of these tend to initiate corrosion that would not occur in their absence, and will accelerate localized corrosion in aggressive environments.

Embedded iron: The surface of stainless steel will pick up particles of carbon steel from steel layout and cutting tables, forming rolls, carbon steel wire brushes, sandblasting, grinding, and from handling with steel slings and clamps. Sheet and plate should be stored in vertical racks to avoid scoring that can take place with floor storage.

Steel particles embedded in the surface of stainless steel will rust when they are exposed to water or a moist atmosphere, showing up as spots and streaks which, if not removed, can produce pitting.

Heat tint: Welding heats the base metal, causing heavy oxide films (scale) to develop in the heat-affected zone (HAZ). These oxide films range in color from light brown to black.

Removing heat tint is costly, and may be un-

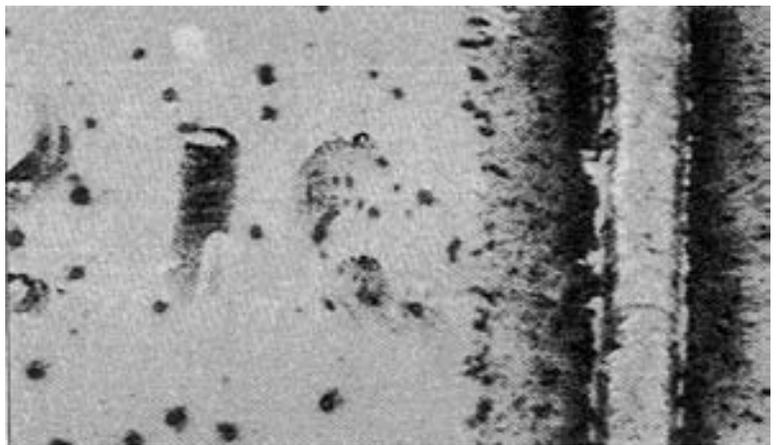
immersion in a 20 to 40% solution of HNO_3 at a temperature of 50 to 60°C (120 to 140°F).

The complete passivation treatment includes degreasing, immersion, and rinsing. Degreasing, preferably in a nonchlorinated solvent, removes organic contaminants from the surface.

Degreasing: Neither air nor nitric acid can form or enhance the protective film when grease, oil, fingerprints, or other organic contamination are present on the surface. Parts must be thoroughly degreased prior to any passivation treatment. The water-break test, described in ASTM A 380, is easy to apply and is effective in detecting residual organic matter that may not have been removed in the degreasing operation. A sheet of water directed over the surface will "break" around oil, grease, and other organic contaminants not completely removed from the surface. Specifications can simply call for no break in the film as it drains from the vertical surface.

Immersion: The part is immersed in a passivating solution selected from ASTM A 380 Table A2.1, Part II or Part III. In addition to the standard HNO_3 solution, there are a number of solution variations appropriate for all grades of 200, 300, and 400 series, maraging, precipitation hardening and free-machining alloys in various heat treat conditions and surface finishes.

Rinsing: Immediate and thorough rinsing in clean water of pH 6 to 8 is mandatory. In many instances neutralization prior to rinsing is help



Rust in the heat-affected zone of this weld on a stainless steel pipe (dark bands on both sides of the weld) was produced by iron embedded in the surface during wire brushing. Rust spots away from the weld were caused by particles from an overhead sandblasting operation that fell on the pipe.

ful. Immersion, neutralization, and rinsing must follow one another without allowing the surface to dry between steps. When passivating stainless steel sheet material, each sheet must be completely dry before it is stacked to avoid water marks.

In addition to the cleaning precautions given in ASTM A 380, different grades of stainless steel should not be mixed in the same passivating bath as this can initiate corrosion where surfaces come in contact.³

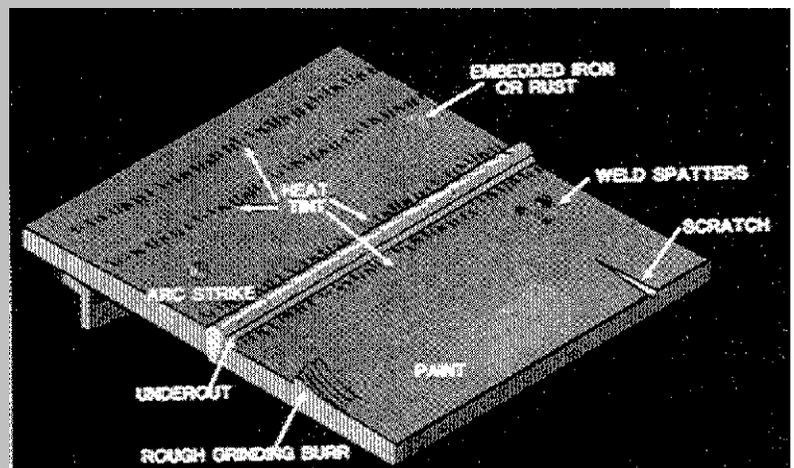
Although nitric acid does not normally corrode

necessary if stainless steel is exposed only to water, alkaline environments, and other mild industrial atmospheres, which seldom initiate corrosion. However, removal of heat tint may be necessary to prevent corrosion in acidic environments, especially when the base metal has very little extra corrosion resistance. In some mild environments, heat tint removal is necessary to prevent contamination of process fluids.

Heat tint must be removed when scrupulously clean surfaces are required to prevent contamination and batch-to-batch carryover of process materials. These applications include evaporators and piping for high-purity and ultrahigh-purity water systems, nuclear piping, pharmaceutical and biological equipment, electronic-chip washing facilities, and brewery equipment.

Weld flux: Weld flux, produced by welding with covered electrodes, is difficult to remove completely, especially from the sides of the weld bead. Wire brushing with stainless steel wire brushes, abrasive disk and flapper wheel grinding, and chipping may leave small flux particles at the side of the bead. These particles are excellent crevice formers, and their complete removal generally requires pickling or electropolishing.

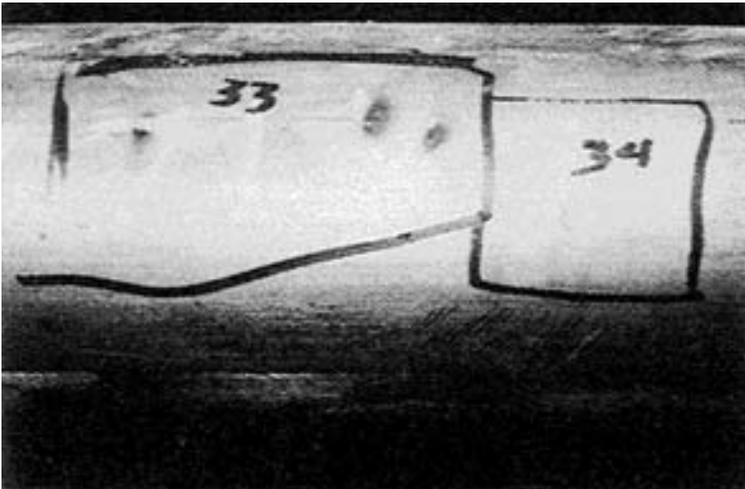
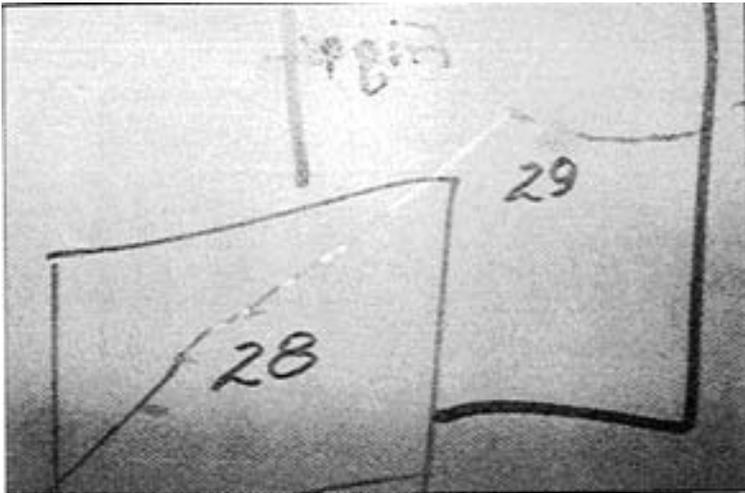
Arc strikes and spatter: Arc strikes produce



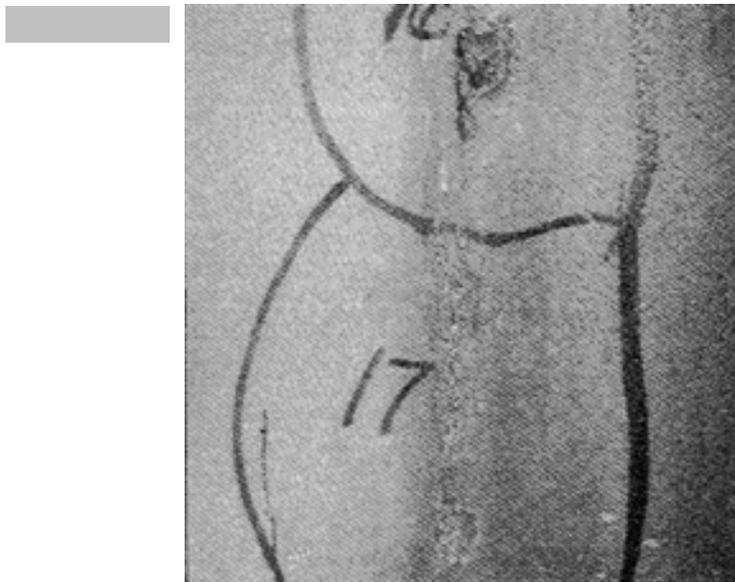
These common surface defects on stainless steel all result from mechanical operations that are used to fabricate components.

small pinpoint surface defects in the protective film, as does weld spatter. If defects cannot be prevented, they should be removed before placing stainless steel into service in aggressive environments.

Scratches and paint: Deep scratches also initiate corrosion, as can paint, crayon marks, and other instruction markings if they are not removed.



Crevice corrosion can be initiated by deep scratches, top and by paint and other markings, such as those made by a marking crayon, bottom. Removal of the markings would have prevented the corrosion.



The heat-affected zone on the opposite side of a weld can also initiate corrosion if the heavy oxide film (scale) is not removed. For example, the corrosion shown here is on the inside of a vessel, in the heat-affected zone of an external weld.

stainless steel, it will corrode surfaces that are significantly altered. Acid cleaning should not be used for carburized and nitrided stainless steel parts nor for improperly heat treated high-carbon/high-chromium martensitic grades that have not been fully hardened.³

Pickling treatments

Passivation treatments are not designed to remove heat tint, embedded iron particles, heat treating scale, and other surface defects produced during fabrication, since nitric acid does not corrode or remove the surface layers having embedded defects. Elimination of these defects requires removal of the normal, protective oxide layer and 25 to 40 μm (0.001 to 0.0015 in.) of the substrate metal by pickling the surface in a nitric-hydrofluoric acid (HNO_3 -HF) bath. The protective film then reforms in air over the freshly cleaned surface. This oxide film is uniform and leaves the stainless surface in its normal passive condition.

While pickling is not strictly a passivating treatment, it provides many of the same benefits. Pickling is most useful for localized cleaning of welded areas, but also can be used to improve the corrosion resistance of mechanically cleaned surfaces.

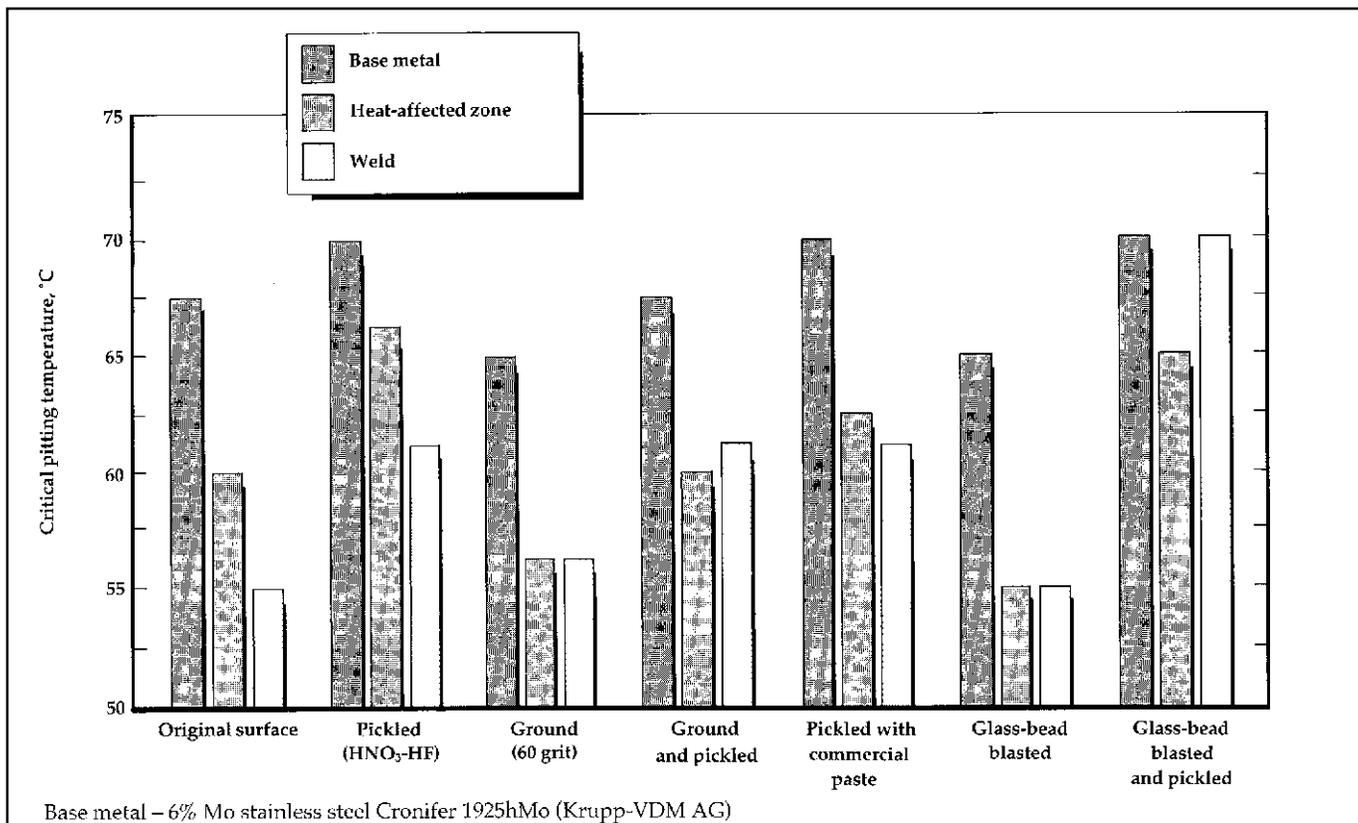
Disposal of pickle liquor is a growing problem that tends to limit pickling by immersion to those fabricators and chemical cleaning contractors who already have pickle tanks and approved arrangements for disposal.

Pickling at the steel mill removes the oxide scale that forms during annealing. Mill pickling also removes manganese sulfides or other inclusions in the surface and removes surface layers that may have been depleted in chromium during annealing.

ASTM A 380 lists three pickling solutions for stainless steel. Fabricated austenitic stainless steels can be pickled by immersion in a standard 10% HNO_3 , 2% HF bath at 50°C (120°F). For local area pickling or if the fabricated component is too large to be immersed, commercial HNO_3 -HF pickle pastes can be just as effective. Pickle paste can be applied with a paint roller or nylon brush.

Paste must be washed off within 15 to 30 min of application, or corrosion will be initiated. Personnel need protective clothing and training in safe handling procedures.

Although post-fabrication pickling improves the performance of stainless steels in a variety of applications, until recently there has been very little research data to support field experience. Quantitative data on the increase in critical pitting temperature in ferric chloride (ASTM G 48) shows that pickling provides a 2.5 to 10°C (4.5 to 18°F) improvement in performance.⁴ While not large, the improvements in lightly ground, and glass-bead blasted surfaces are uniformly positive, indicating that pickling provides benefits beyond those obtained with the best controlled mechanical treatments.



Pickling of stainless steel increases the critical pitting temperature in ferric chloride (per ASTM G 48) in the base metal, heat-affected zone, and weld areas. Mechanical cleaning treatments that are performed without a succeeding pickling treatment decrease the critical pitting temperature.



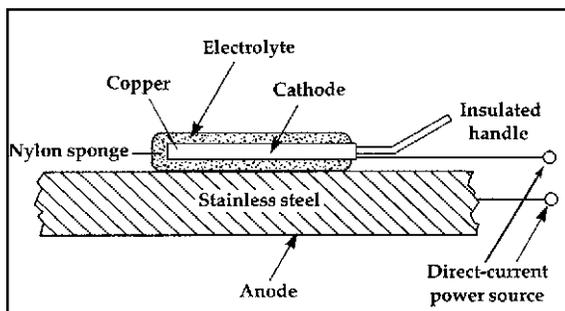
Pickling removes heat tint and embedded surface contaminants.

Electrocleaning and electropolishing

Electrocleaning, an electropolishing technique, is a useful alternative to pickling treatments. Although electrocleaning is not covered under ASTM A 380, it is widely used to remove imperfections from the surface of stainless steel after fabrication. It removes embedded iron particles and similar film defects as does pickling. Unlike pickling, electrocleaning does not roughen the surface, but makes it smoother. A 12-Vdc power source with variable current capability is connected to the stainless steel, making it the anode. A copper cathode and an electrolyte – usually phosphoric acid – are then used to corrode away the protective film and several layers of the surface in a controlled manner by varying the current and dwell time.

Electrocleaning can be performed in most plating shops by immersion. Localized electrocleaning with field kits is widely practiced to remove heat tint and weld-related defects from the heat-affected zone.

Electropolishing is the same process as electrocleaning, but is generally performed for a longer time. It is used to polish large surfaces. One use is the polishing of pulp mill headboxes to prevent pulp hang-up on tiny surface imperfections. It is also used in the electronics and biotechnology industries to clean and smooth the inside surfaces of pipe and tubing. Large nuclear power plant components also are electropolished to refurbish contaminated surfaces.



Localized electrocleaning of heat tint from the surface of stainless steel can be performed with a simple tool such as the one shown here.

Electropolishing, because it uses milder acids and can be performed locally, reduces the volume of waste liquor for disposal compared with immersion pickling. However, personnel must be protected and disposal regulations must be observed, as is done in other pickling, passivating, and electrocleaning operations.

Since both pickling and electropolishing remove metal, neither process can be used on polished surfaces without altering the surface finish. Care should also be used when pickling or electropolishing machined surfaces. Although only about 25 μm (0.001 in.) of the surface is corroded away, this may be enough to alter tolerances on some closely dimensioned parts.

Mechanical cleaning

Section 5.3 of ASTM A 380 describes mechani-

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cal descaling methods commonly used to clean welds. These include abrasive blasting, brushing, grinding, and chipping. However, if these mechanical cleaning procedures are not performed carefully, they can do more harm than good.

Grit blasting can be extremely detrimental as it is almost impossible to prevent particles of grit from becoming embedded in the surface being blasted. Grit blasting also roughens the surface to the point where crevice corrosion becomes likely. Peening with clean stainless steel shot, which produces compressive stresses in the surface, reduces the risk of stress corrosion cracking in some applications. However, this must be balanced against the increased risk of crevice corrosion due to the roughened surface.

Sand blasting should be avoided unless no other cleaning method can be used. This is often the case when cleaning stainless steel tank bottoms that have been inadequately protected from contamination during construction. Only new, uncontaminated sand should be used, and then only once.

Blasting with clean glass beads is an effective method for local- and large-area cleaning. Clean walnut shells also are a useful blasting medium.

Grinding with clean aluminum oxide disks or clean flapper wheels can be used to remove heat tint and other weld-related defects. However, even light grinding leaves a cold worked,

smearing surface that has microcracks, laps, seams, and other sites that can initiate crevice corrosion in aggressive environments. Heavy grinding with grinding wheels overheats the surface of stainless steel and degrades its corrosion resistance to depths greater than the 25 to 50 μm (0.001 to 0.002 in.). As a result, grinding should be used only when removal of the weld reinforcement (weld crown) is more important than optimizing corrosion resistance.

Chipping is normally used between weld passes to remove weld slag, and subsequent weld passes normally eliminate any damaging effects.

Inspection procedures

Several methods of evaluating cleanliness after fabrication are described in ASTM A 380. The water-break test described previously is used to determine whether organic contamination has been removed from the surface. Water also is useful in detecting iron contamination: rust streaks and spots will form on wetted surfaces over a period of several hours if contamination is present.

The copper sulfate and ferroxyl tests are much more sensitive than the water test, and are specified when the surface must be entirely free of iron. Special considerations apply when testing equipment intended for use with food, beverages, or other products for human consumption. The ferroxyl test is effective and easy to use, although the solution does not have a long shelf life. ■

For more information: Contact Mr. Arthur H. Tuthill, P.E., at Tuthill Associates Inc., P.O. Box 204, Blacksburg, VA 24060 (tel: 703/953-2626; fax: 703/953-2636), or Mr. Richard E. Avery at Avery Consulting Associates Inc., 117 Winterwood Dr., Londonderry, NH 03053 (tel: 603/434-2625; fax: 603/425-2542).

Information on stainless steels can be obtained from the Nickel Development Institute, 214 King St. W. Suite 510, Toronto, Ontario, Canada M5H 3S6 (tel: 416/591-7999; fax: 416/591-7987). Copies of ASTM A 380-88 can be ordered from the American Society for Testing and Materials (ASTM), 1916 Race St., Philadelphia, PA 19103-1187 (tel: 215 / 299-5400; fax: 215 / 977-9679).

References

1. "Cleaning and Descaling Stainless Steel Parts, Equipment and Systems": ASTM A 380-88, American Society for Testing and Materials Philadelphia, Pa.
2. "An X-Ray Photo-electron Spectroscopic Study of Surface Treatments of Stainless Steels," by K. Asami and K. Hashimoto: *Corrosion Science*, Vol. 19, 1979, p. 1007-1017.
3. "Passivation of Stainless Steel Parts," by T. DeBold: *TAPPI Journal*, January 1988, p.196-198.
4. Dr. Rockel, Krupp VDM GmbH, private communication.