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PASSIVATION OF STAINLESS STEEL

Getting the Properties You've Paid For

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The conversation usually starts out something like this: “Hey, this is Joe from Joe's Machine Shop. We've got a job in here we're working on and the customer wants us to have some kinda passivate coating something-or-other. You guys do that? How thick is that stuff? Is that like some kinda plating or paint or something? What color is it? How much tolerance should I allow for it?” Usually, the opening statement ends with a phrase like, “I don't even know why they need it. What's the point of using stainless steel if you're gonna put some kinda coating on it anyway?”

The fact of the matter is, “Joe” is not the exception. Many machine shops, purchasing agents, and engineers alike are somewhat in the dark when it comes to the relationship between corrosion resistant (stainless) steel and chemical passivation. Even among the metal finishing community, there is some disagreement about the theory behind the process of chemical passivation. Some believe it is effective because it is a cleaning process. Others credit the enhanced corrosion resistant properties to the thin, transparent oxide film resulting from chemical passivation. Regardless of the argument, the bottom line is, it works! Verification tests, including copper sulfate immersion, and accelerated corrosion tests, such as salt spray, high humidity, and water immersion, undisputedly confirm the effectiveness of chemical passivation. Advanced material engineers in aerospace, electronics, medical, and similar high-tech industries have utilized chemical passivation for many years. Their applications demand the maximum performance from components manufactured from corrosion resistant (stainless) steels, and they realize that passivation is one of the most effective methods of achieving the desired results.

What is Passivation?

According to ASTM A 380, passivation is “the removal of exogenous iron or iron compounds from the surface of a stainless steel by means of a chemical dissolution, most typically by a treatment with an acid solution that will remove the surface contamination but will not significantly affect the stainless steel itself.” In addition, it also describes passivation as “the chemical treatment of a stainless steel with a mild oxidant, such as a nitric acid solution, for the purpose of enhancing the spontaneous formation of the protective passive film.”

In layman's terms, the passivation process removes “free iron” contamination left behind on the surface of the stainless steel as a result of machining and fabricating processes. These contaminants are potential corrosion sites which, if not removed, result in premature corrosion and ultimately result in deterioration of the component. In addition, the passivation process facilitates the formation of a very thin, transparent oxide film, which protects the stainless steel from “selective” oxidation (corrosion). So what is passivation? Is it cleaning? Is it a protective coating? In my opinion, it is a combination of both!



How is the Passivation Process Performed?

The process typically begins with a thorough cleaning cycle. It is intended to remove oils, greases, forming compounds, lubricants, coolants, cutting fluids, and other undesirable organic and metallic residue left behind as a result of fabrication and machining processes. General degreasing and cleaning can be accomplished by a variety of commonly accepted methods, including vapor degreasing, solvent cleaning, and alkaline soaking. After removal of the organic and metallic residues, the parts are placed into the appropriate passivation solution. Although there are many variations of passivating solutions, the overwhelming choice is still the nitric acid based solutions. Recently, there has been substantial research performed to develop alternative processes and solutions that are more environmentally friendly, yet equally effective. Although alternative solutions containing citric acid and other types of proprietary chemistry are available, they have not been as widely accepted commercially as nitric acid based solutions.



TWO-HOUR salt spray test per ASTM B-117
(With passivation on the left and without on the right.)

The three major variables that must be considered and controlled for the passivation process selection are time, temperature, and concentration. Typical immersion times are between 20 minutes and 2 hours. Typical bath temperatures range between room temperature and 160°F. Nitric acid concentrations in the 20% to 50% by volume range are generally specified. Many specifications include the use of sodium dichromate in the passivation solution, or as a post passivation rinse, to aid in the formation of a chromic oxide film. Careful solution control, including water purity, ppm (parts per million) of metallic impurities, and chemical maintenance, is crucial for success.

The type of stainless steel being processed is the determining factor when selecting the most effective passivation process. Bath selection (time, temperature, and concentration) are all a function of the type of alloy being processed. A thorough knowledge of the material types and passivation processes is paramount to achieving the desired results. Conversely, improper bath and process selection and/or process control will produce unacceptable results, and in extreme cases, can lead to catastrophic failure, including extreme pitting, etching and/or total dissolution of the entire component.



Equipment and Precautions

Passivation should only be performed by trained, experienced technicians familiar with the potential hazards associated with the science. Safety practices must be fully understood when handling passivation chemicals. Special boots, gloves, aprons and other safety equipment must be utilized. Tanks, heaters and ventilation, as well as baskets and racks, must be appropriately engineered to perform the process. Iron or steel parts or equipment must never be introduced to the process, or the results can be devastating! Furthermore, in order to comply with EPA requirements, the necessary water and air permits and treatment capabilities must be in place. The days of the “mom-and-pop” shops performing passivation in a stone crock in the back corner of the shop are diminishing, due to safety and environmental concerns.

Specifications and Verification Testing

There are a few generally accepted industry specifications available for reference when choosing a passivation process. They offer time, temperature, and concentration information, and subsequent testing requirements to validate the effectiveness of the process. Many large corporations have developed internal specifications to control their unique requirements regarding passivation and verification testing. Regardless of the situation, it is usually prudent to reference a proven procedure when requesting passivation. By referencing a specification, you don't have to “reinvent the wheel,” and by taking advantage of the past experiences of others, both successes and failures, you can eliminate much of the guesswork that would otherwise accompany a new process.

Although recently cancelled, the most commonly referenced industry specifications regarding passivation are Fed. Spec. QQ-P-35C, which is now superseded by ASTM A-967, and ASTM A-380. All three are well written, well defined documents which provide guidance on the entire process, from manufacturing to final testing requirements. If you're not sure what you need, they can be referenced in full, or selectively. The testing requirements can be utilized or waived, depending upon the individual situation. One of the most commonly specified verification tests is the copper sulfate test. Passivated parts are immersed in a copper sulfate solution for 6 minutes, rinsed, and visually examined. Any copper (pink) color indicates the presence of free iron, and the test is considered unacceptable. Other validation tests include a 2 hour Salt Spray or 24 hour high-humidity test. These tests are performed by placing passivated parts in a highly controlled chamber which creates an accelerated corrosive environment. After subjecting the test pieces to the corrosive atmosphere for the prescribed exposure periods, the parts are removed and evaluated. Although results can be somewhat subjective, ASTM B-117 is an excellent reference in determining acceptability. It is important to note that each of the test methods mentioned have different advantages and limitations. Care should be taken to select the appropriate test methods, based on alloy type and end use environment.



Machining and Heat Treating Techniques

Perhaps the most overlooked variable in the entire passivation equation is the negative impact of poor machining and heat treating practices. All too often, gross contamination introduced during manufacturing and/or thermal processes leads to unacceptable test results. The following practices will reduce gross contamination during manufacturing and increase the chances of successful passivation and test results:



Stainless steel parts are supposed to be
rust - resistant.

So how does this happen?

- Never use grinding wheels, sanding materials, or wire brushes made of iron, iron oxide, steel, zinc, or other undesirable materials that may cause contamination of the stainless steel surface.
- The use of carbide or other non-metallic tooling is recommended whenever possible.
- Grinding wheels, sanding wheels, and wire brushes that have been previously used on other metals should not be used on stainless steel.
- Use only clean, unused abrasives such as glass beads or iron-free silica or alumina sand for abrasive blasting. Never use steel shot or grit, or abrasives which have been used to blast other materials.
- Thorough cleaning prior to any thermal processing is critical! Stress relieving, annealing, drawing, or other hot-forming processes can actually draw surface contaminants deeper into the substrate, making them almost impossible to remove during passivation.
- Care should be taken during all thermal processes to avoid the formation of discoloration (oxides). Passivation is not designed to remove discoloration, and will not penetrate heavy oxide layers. In extreme situations, additional pickling and descaling operations are required prior to passivation to remove the discoloration.
- Controlled atmosphere ovens are highly recommended for all thermal processes to reduce airborne contamination and prevent oxides from developing.



Conclusion

So how do you get “the performance you've paid for” from high-dollar stainless steel alloys? It really boils down to a basic understanding that the passivation process is both an art and a science, and that machining, fabricating, and heat treating practices can substantially affect the corrosion resistance of the component. It's a well-known fact that passivation will enhance the corrosion resistance of stainless steels, but to realize the maximum performance from these high-tech alloys, all parties involved with manufacturing must understand their responsibility in maintaining the integrity of the material throughout the process.



Copper Sulfate test per MIL-STD-753.
(With passivation on left and without on right.)

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References

1. ASTM A 380 – 96: Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems
 - ASTM Committee A-1 on Steel, Stainless Steel, and Related Alloys
 - ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428

2. Fed. Spec. QQ-P-35C, October 28, 1988: Passivation Treatments For Corrosion Resistant Steel
 - CANCELLED April 4, 1997
 - SUPERSEDED by ASTM A 967 – 96

3. ASTM A 967 – 96: Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts
 - ASTM Committee A-1 on Steel, Stainless Steel, and Related Alloys
 - ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428

4. ASTM B 117 – 95: Standard Practice for Operating Salt Spray (Fog) Apparatus
 - ASTM Committee G-1 on Corrosion of Metals
 - ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428