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METAL FINISHING

PASSIVATION OF STAINLESS STEEL

The conversation usually begins like this: "Hey, this is Joe from Joe's Machine Shop. We have a job in here and the customer wants us to have some kind of passivate coating something or other. Do you guys do that? How thick is that stuff? Is that like plating, paint or what? What color is it? How much tolerance should I allow for it?" The opening statement usually ends with a phrase like; "I don't even know why they need it. What is the point of using stainless steel if you are going to put some kind of coating on it anyway?"

Joe is not the exception. Many machine shops, purchasing agents and engineers are somewhat in the dark when it comes to the relationship between corrosion resistant (stainless) steel and chemical passivation. Even among the 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 resistance properties to the thin, transparent oxide film resulting from chemical passivation. Regardless, the bottom line is that 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 passivation for years. The applications demand the maximum performance from components manufactured from-corrosion-resistant steels, and they realize that passivation is one of the most effective methods of achieving these results.

WHAT IS PASSIVATION?

According to ASTM A380, passivation is "The removal of exogenous iron or iron compounds from the surface of 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 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 lay terms, the passivation process removes "free iron" contamination left behind on the surface of the stainless steel from machining and fabricating. These contaminants are potential corrosion sites ultimately result in deterioration of the component if not removed. In addition, the passivation process facilitates the formation of a thin, transparent oxide film that protects the stainless steel from selective oxidation (corrosion). So what is passivation? Is it cleaning? Is it protective coating? It is a combination of both?

How is passivation performed? The process typically begins with a thorough cleaning cycle. It removes oils, greases, forming compounds, lubricants, coolants, cutting fluids and other undesirable organic and metallic residue left behind because of fabrication and machining processes. General degreasing and cleaning can be accomplished many ways, including vapor degreasing . solvent cleaning and alkaline soaking.

After removing organic and metallic residues, the parts are placed into the appropriate passivation solution. Although there, are many variations of passivation 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.

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 min and two hours. Typical bath temperatures range between room temperature and 160° F. Nitric acid concentration in the 20% to 50% by volume range is 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 of metallic impurities and chemical maintenance, are crucial for success.

The type of stainless steel determines the most effective passivation process. Bath selection (time, temperature and concentration) is a function of the type of alloy 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. In extreme cases, this can lead to catastrophic failure, including extreme pitting, etching and/or total dissolution of the entire component.

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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 used.

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 mom-and-pop shops performing passivation in a stone crock in the back of the shop are gone.

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 do not have to reinvent the wheel. By taking advantage of the experiences of others, both successes and failures, you can eliminate much of the guesswork that would otherwise accompany a new process.

Although recently canceled, the most commonly referenced industry specifications regarding passivation are Federal Specification QQ-P-35C, which is now superseded by ASTM A-967 and ASTM A-380. All are well-written, well-defined documents that provide guidance on the entire process, from manufacturing to final testing requirements. If you are not sure what you need, they can be referenced in full or selectively. The test requirements can be used or waived, depending on the individual situation.

One of the most commonly specified verification tests is the copper sulflate test. Passivated parts are immersed in a copper sulfate solution for six minutes, rinsed and visually examined. Any popper (pink) color indicates the presence of free iron and the test is considered unacceptable.

Other validation tests include a two-hour salt spray or 24-hour high humidity test; these tests are performed by placing passivated parts in a highly controlled chamber that 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-1 17 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, cross contamination introduced during manufacturing and/or thermal processes leads to unacceptable test results. The following practices will reduce cross contamination during manufacturing and increase the chances of successful passivation and tests results.

- 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.

- 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, grit or abrasives that 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 oxides. Passivation is not designed to remove discoloration and will note 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.

So how do you get the performance you have paid for from high-dollar stainless steel alloys? It 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. 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.

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