Improvement of First Run Pass Rates for Copper-Containing Stainless Steel Components

Technical Experience Brief

Author

Jim Mattzela

Solution Team

Mark Lawrence, Sales Manager
Greg Long, Mfg. Manager
David Misera, Quality Manager
Paul Hoover, Mfg. Technician
Min Yuan, R&D Scientist
Jim Mattzela, R&D Scientist

Abstract

The SilcoTek® manufacturing and quality teams noticed excessively low first run pass rates when coating sampling componentry from a particular customer who manufactures instrumentation for analyzing trace-level volatile and semi-volatile organic compounds (VOCs and SVOCs). The high costs of re-working the parts several times presented a problem to both SilcoTek and the customer, requiring the formation of a comprehensive team to determine the root cause and solution to the problem.

In order to determine the fundamental nature of the surface contaminant that made these parts difficult to process, the R&D team tested several surface preparation methods. They determined that copper was the primary contaminant following a thorough analysis of results from nitric acid-soaking the stainless steel parts. The manufacturing team then standardized a process for removing the copper contaminant from the surface of the parts prior to the CVD coating process which has shown outstanding performance over multiple batches.

Lit. Cat. #EL-001

October, 2014

Problem: Some customer parts, specifically these sampling components we'll call "Part A" and "Part B," yield an unacceptable visual pass rate through standard manufacturing processes, i.e. surface preparation and SilcoNert® coating procedures.

Introduction

Although SilcoTek's chemical vapor deposition (CVD) coating process is compatible with many different substrates, there are some - especially copper - that cannot be properly coated even if they have the required thermal stability (the Silco'd process goes as high as 450° C). The amorphous silicon base layer of all the Silco- and Dursan® coatings does not adhere properly to copper-containing substrates. Rather than uniformly growing across the entire substrate, the coating adheres inhomogeneously on the surface and grows vertically. This leads to easy flaking and an appearance that is uncharacterstic of SilcoTek's line of coating solutions.

It's easy to spot a substrate primarily comprised of copper due to it's red-brown color, but visually detecting contamination is impossible when what seems to be typical 300 grade stainless steel contains trace copper from shared manufacturing processes. The solution team's investigation shows that even "pure" stainless steel components that were manufacturered in the same facility as copper-containing substrates (e.g. brass, Monel®) are susceptible to coating issues due to cross-contamination between processing tools, cutting oils, lubricants, etc. The following information details the steps of the solution process.

Background

We reviewed the historical pass rates for the two part numbers over approximately the past two years. The poor pass rates were, at the time, attributed to dirty and unclean appearance post-coating which prompted several re-work cycles (as many as six) to get them up to SilcoTek's quality standards.

On a couple of occasions, resurfacing the parts was necessary in order to remove surface blemishes which were not affected by our standard operating procedure (SOP) caustic surface preparation techniques.

Multiple rework cycles are costly to SilcoTek both in terms of direct costs of labor and machine time as well as shipping delays to customers. By working with the customer directly, the entire group concluded that the current process for coating the subject parts was unacceptable and therefore a process improvement task team was instituted. Figure 1 on the next page shows the pass rate for Part A (Fig. 1a) and Part B (Fig. 1b).

TIP: To see a list of all the materials and substrates that are compatible/incompatible with SilcoTek's chemical vapor deposition (CVD) coating process, please click here or call 814-353-1778 (2 at the operator) to speak to a technical representative.

®Monel is a registered trademark of Special Metals Corporation

Part A

Part B

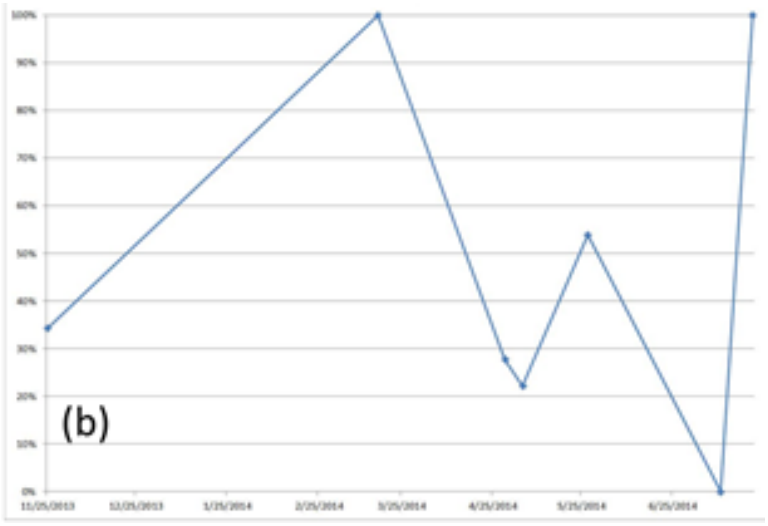

Figure 1 - Pass rate summary for copper-contaminated part A (a) and part B (b) from late 2013 to July 2014

Experimental

Our initial hypothesis was that some surface contaminant was not being effectively removed by the SOP caustic method. Upon receiving a large batch of parts, several experimental pathways were outlined to establish data based on a variety of surface preparations. Pieces were routed to go through: SOP caustic (control), Safety-Kleen® (mineral spirits) followed by SOP caustic, EnTron™ (n-propyl bromide) followed by SOP caustic, hexanes and isopropanol followed by SOP caustic, and nitric acid followed by SOP caustic. Nothing unusual was noted during any of the solvent pre-clean experiments (Safety-Kleen, Entron, or hexanes+IPA). During the nitric acid soak (10 minutes in 10% mix of nitric acid with deionized water), a dark cloud developed around the parts in the solution (Figure 2).

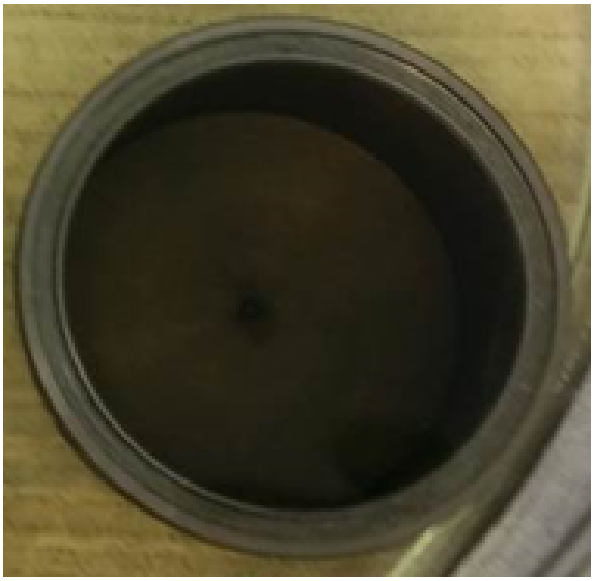

Figure 2 - Part B in nitric acid after 10 minutes of soak time. Discoloration appears due to surface contamination (copper).

Results & Discussion

We removed the parts from the solution and noted what appeared to be fine particulate on the surface. It was possible to wipe the particulate from the part surface, but some ‘staining’ on the surface was still noted. At this point, several pieces had been coated. None of these pieces passed QC specifications, but the nitric acid-treated parts did not exhibit the "typical" failures, though the staining was still visible through the coating.

In the morning following the nitric acid soak, our team noticed that the nitric acid solution had turned bluish in color. With some quick experimentation, we discovered that copper dissolved into nitric acid would also create a blue-colored solution. The team investigated this further via two methods: for a control, we dissolved a pre-1982 penny in nitric acid solution and then performed a "flame" test to search for copper ions in the solution. The dissolved penny showed a bright blue color - much brighter than the solution used to process parts A and B. A flame test provided a second verification as the copper ions would "burn" green, which indeed happened.

Further nitric acid testing showed that the contaminant (suspected to be copper) was still on the Safety-Kleen®-prepped population as well as the control group - none of which had been coated. Coated pieces that failed QC were also tested and we detected very little solution discoloration. The manufacturing team had to perform multiple re-works yet again in order to get the parts to an acceptable appearance.

When we received a subsequent batch that hopefully was free of contamination, we pulled one part B for SEM/EDS analysis. The team immediately contacted the customer to discuss possible contamination since we detected a small amount of copper once again. Then, a breakthrough: the customer mentioned that their part manufacturer also processed brass at their facility. Brass is comprised of copper and zinc, both of which are incompatible with the Silco'd coating process.

We further refined the nitric acid method to yield a processes which showed repeated success.

1. Soak parts in fresh nitric acid solution for 10 minutes
2. Rinse in deionized water and hand wipe to remove the bulk of copper residue
3. Return to nitric acid "upside-down" for 10 more minutes, then repeat step 2
4. Resurface parts through standard techniques (lathe, sandpaper, Scotch-Brite®)
5. SOP caustic cleaning/surface preparation

This was, of course, a time-consuming solution, but the method yielded a success rate of 17 for 17 pieces over three separate processing batches.

Conclusion

Nitric acid was successfully used to detect an unknown surface contaminant on customer's sampling components. The contaminant was determined to be copper in this instance. We developed a special surface preparation method to yield acceptable first run pass rates.

Although this method works, it is not an efficient long-term solution as detailed above. SilcoTek® has partnered with this particular customer to explore copper-free sources for manufacturing their parts.

Figure 3 - A "to -spec" part successfully coated without copper issues

Stainless steel is the best substrate for SilcoTek's CVD coating process, although other metals, glass, and ceramics can also be coated with equal success. To ensure that your next shipment of parts for coating is processed quickly and properly, please contact our sales team prior to sending them to SilcoTek's facility by sending an email to SilcoD@SilcoTek.com. They will review your part photos/drawings and confirm that everything you want to send can be coated properly. If there are any red flags, we will work with you to find a solution without disrupting your business or its valuable supply chain workflow.

SilcoTek®
Driving Innovation