



# Iodine Vapor Exposure Testing of SilcoTek Coatings\*

## Technical Insight

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\* This Technical Insight is a summary of a technical report provided by the NASA Glenn Research Center; Cleveland, OH.

Please see last page for a full citation of this work.

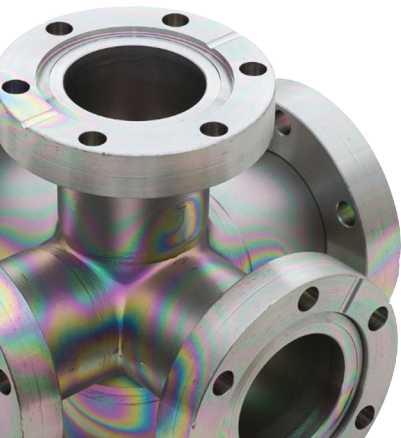
### Background

Hall-effect thrusters have been used in satellites and medium-sized robotic space vehicles since the 1970s. These thrusters work by ionizing a propellant and accelerating these ions with an electrostatic potential to produce thrust. Xenon gas is the most commonly used propellant due to its high atomic weight (easy to ionize) and its chemical inertness; however, storage requires low temperatures and high pressures. NASA Glenn Research Center has demonstrated that Hall-effect thrusters operating with iodine as the propellant can perform at the same thrust and efficiency compared to state-of-the-art xenon thrusters.

Iodine has many logistical benefits over xenon for use in these thrusters. Iodine stores at 3 times the density of xenon, it can be stored at less than 15 psi, whereas xenon requires 2500 psi, and iodine is much more abundant than xenon, and therefore cheaper. Despite these benefits, there are challenges to consider with material compatibility. Very little is known regarding iodine reactivity with the wide range of spacecraft materials in a vacuum environment. This information would also be useful to the nuclear industry as iodine is regarded as one of the most hazardous fission products from a fuel nuclear reactor. NASA has developed an experiment to study the interaction between iodine and a variety of common spacecraft materials. In their tests, they have included Silcolloy® and Dursan®-coated coupons for evaluation.

### Goal

Quote taken from NASA's report:



“The goal of this work is to begin studying the chemistry, structure and microstructural, and kinetic properties of materials and coatings exposed to iodine in a relevant environment to spacecraft technology development. For the study described in this report, five common spacecraft and test facility metals and two coatings were selected. The results of this work are pertinent to vacuum facilities and some regions of space since these environments contain molecular or atomic oxygen at very low levels.”

This report investigated iodine exposure to 304 and 316 stainless steel, A36 low carbon steel, 6061 aluminum, and 6Al-4V TiAl alloy. The study also includes Silcolloy coated on every alloy except for the 6061 aluminum, and Dursan only on a 304 stainless steel substrate.

## Results and Discussion

The iodine exposure was done in a tubular furnace that was held at 300°C and flushed with 145 mL/min of argon gas along with 1 mg/min of iodine vapor. The testing exposed the coupons in triplicate for 5, 15, and 30 days. The coupons were monitored via weight measurements, XRD, and SEM. Weight change can be broken into three distinct groups: the steels which gained weight (Figure 1), the aluminum alloys which lost weight (Figure 2), and the coated coupons that had no weight loss or gain except for Silcolloy on 304 stainless steel which gained weight past the 15-day measurement period (Figure 3).

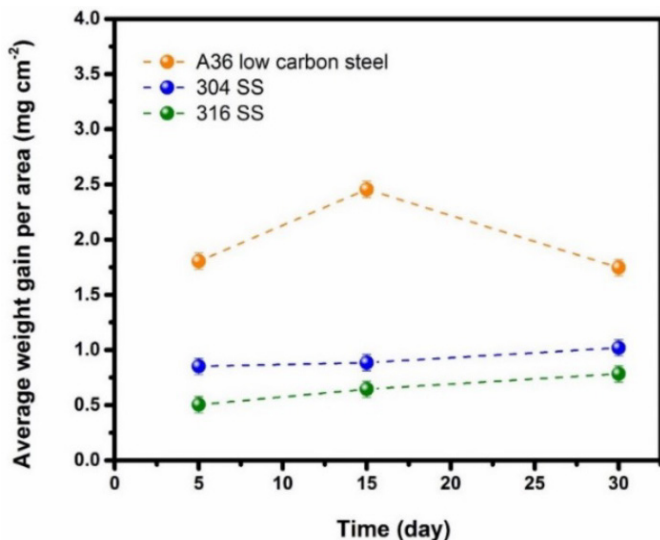


Figure 1: Weight gain per area vs. time for bare steels.

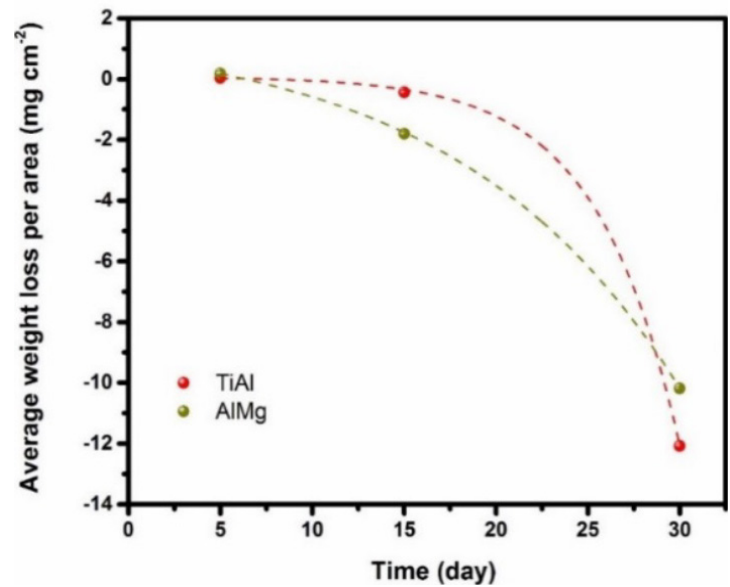


Figure 2: Weight loss per area vs. time for bare aluminum alloy.

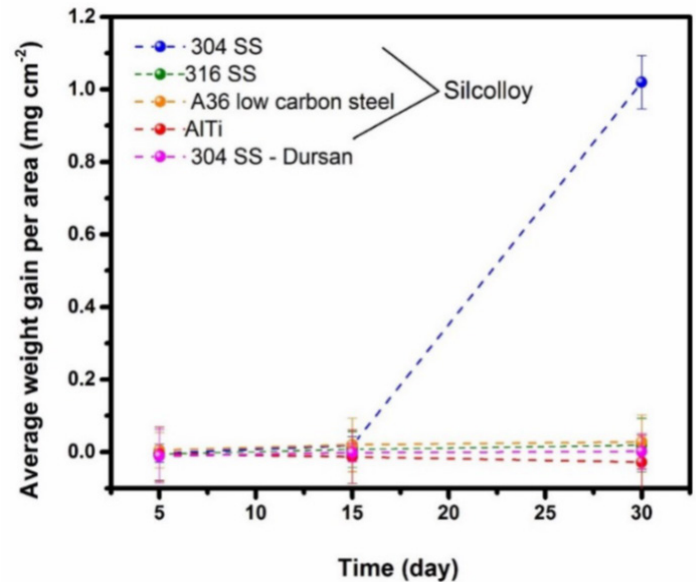


Figure 3: Weight of SilcoTek-coated coupons. Silcolloy on 304SS is the only coupon to show any appreciable weight change.

XRD and SEM analysis shows that the weight gain measured in the steel samples is due to oxidation of the surface. The authors explain that measures were taken to exclude oxygen from the testing environment, it one possible source is could be when the iodine was introduced to the system as a switch from one Erlenmeyer flask to another was needed to start the flow of iodine vapor. Another possible source is from the argon tank used for inert flow in the system. Even at high purity levels, argon tanks are known to have oxygen at ppm levels, which would be enough to cause the oxidation seen in these experiments.

A cross section of a 316 stainless steel sample prior to and after iodine exposure is shown in Figure 4. The scale on the surface after exposure is approximately 3.8  $\mu\text{m}$  thick and not well bonded to the surface. XRD showed that the pure austenite surface prior to exposure reacted to form hematite ( $\text{Fe}_2\text{O}_3$ ) and nickel chromium iron oxide ( $\text{NiCrFeO}_4$ ). These results are in good agreement with previous studies that suggest iodine will catalyze oxidation of stainless steel even at room temperature in air.

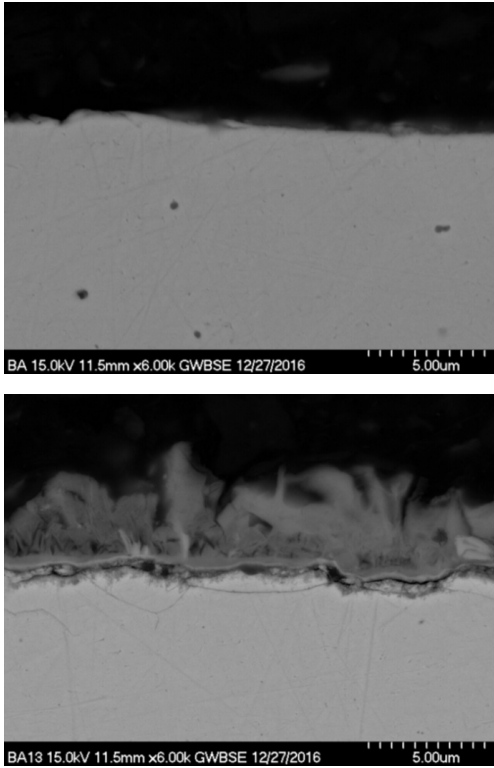


Figure 4: Backscatter electron images of the cross-section of 316 stainless steel prior to exposure and after 30 days of exposure.

The two aluminum alloys underwent weight loss as a result of the iodine exposure. Figure 5 shows a cross section of the TiAl alloy before and after exposure. There is no evidence of scale formed on the surface of the exposed alloy, and a yellowish deposit was found on the inner walls of the tubular furnace. When this deposit was analyzed with EDS it was found that it was composed of titanium, aluminum, iodine, and oxygen. This suggests that the iodine reacted with the surface and formed volatile compounds that were then removed from the surface due to the flowing nature of the experiment.

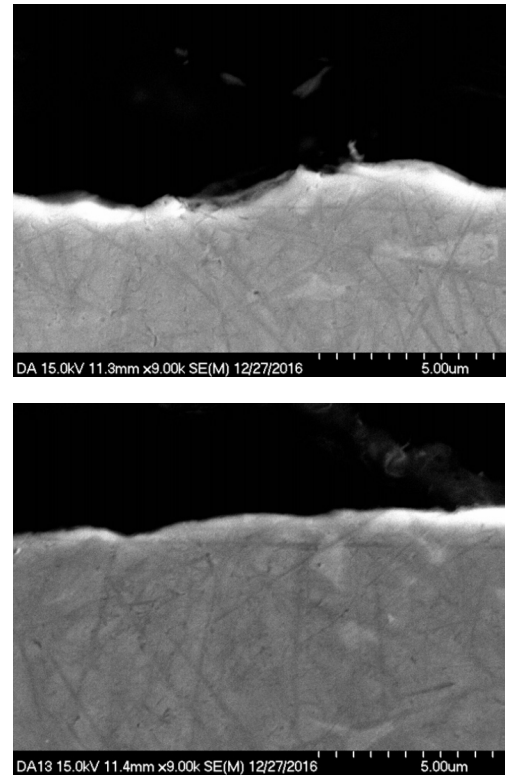
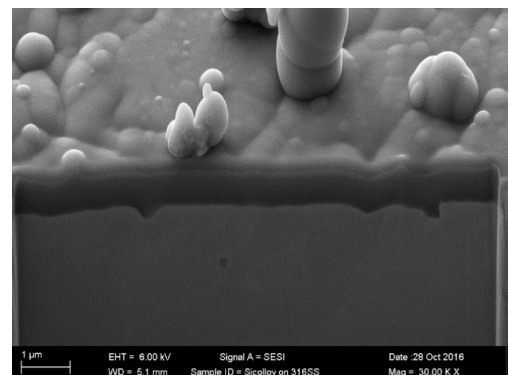
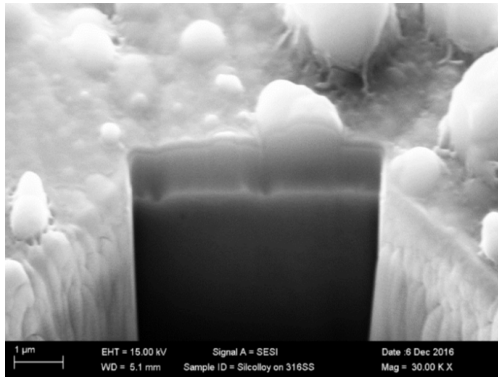


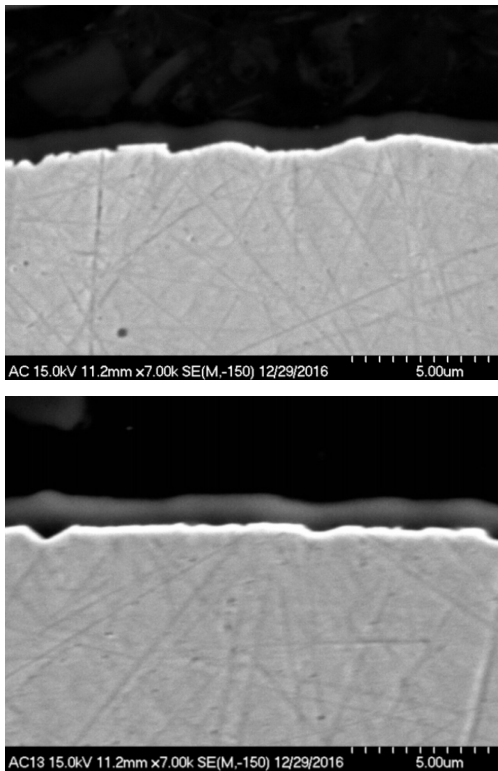
Figure 5: SEM of the cross-section of the TiAl alloy before and after 30 days of exposure to iodine.

The coated samples did not change weight after the 30-day exposure except for the Silcolloy coated 304 stainless steel. Figures 6 and 7 show the cross-section of a Silcolloy coating on 316 stainless steel and Dursan on 304 stainless steel, respectively. There was no chemical attack of the steel surface in either case; however, EDS analysis does show that the Silcolloy coating had increases in oxygen (most evident in the coating of 304 steel which may be the assignable cause to the weight gain), iron in the steel coated samples, and both titanium and aluminum in the TiAl samples. Dursan-coated steel showed little to no elemental change after exposure.





**Figure 6:** FIB cross-section of 316SS coated with Silcolloy, before (top) and after (bottom) exposure to iodine vapor. Little to no change in the coating or substrate was observed.



**Figure 7:** SEM of a polished cross-section of Dursan-coated 304SS before (top) and after (bottom) exposure to iodine vapor. Little to no change in the coating or substrate was observed.

XRD analysis of the Silcolloy and Dursan on steel coupons showed only austenite and a small amount of iron silicide ( $\text{Fe}_{0.95}\text{Si}_{0.05}$ ) prior to and after iodine exposure. The TiAl coupon with Silcolloy showed TiAl and titanium suboxides prior to exposure, and the same after exposure with the addition of the anatase titania phase ( $\text{TiO}_2$ ). The authors did not comment on this change in the coated samples, so it is unclear as to whether this would be a benefit or harmful to the substrate.

## Conclusion

The authors concluded that Silcolloy® and Dursan® were effective in protecting the coupons that were studied. The only outlier was Silcolloy on 304 stainless steel. The coating on the 304 SS was much thinner than the Silcolloy coating on the other steels (~500nm on 304 SS whereas the 316 SS had a ~1000 nm Silcolloy coating). The authors suspect that the failure was related to the surface preparation before the coating was deposited since it worked so well on all other substrates. They plan to investigate further to address the anomaly.

\*Thank you to the authors for providing SilcoTek® with this valuable research.

Costa, G.C.C., Benavides, G.F., and Smith, T.D. “Chemical, Structural, and Microstructural Changes in Metallic and Silicon-Based Coating Materials Exposed to Iodine Vapor” May 1, 2017 Technical Report. NASA Glenn Research Center; Cleveland, OH.



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