ANL/CSE-13/22

Evaluation of Valves After Concomitant Irradiation and Contact with Flowing pH 1 Sulfuric Acid

Chemical Science and Engineering Division

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EVALUATION OF VALVES AFTER CONCOMITANT IRRADIATION AND CONTACT WITH FLOWING PH 1 SULFURIC ACID

Several valves have been tested at the Argonne National Laboratory (Argonne) 3.0 MeV Van de Graaff generator. Valves have been tested with either flowing nitric acid (HNO_3) or sulfuric acid (H_2SO_4). This brief summary will focus on those valves that were exposed to H_2SO_4.

Three valves were exposed to high-energy x-rays generated from a tungsten converter that was irradiated by a –2.5 MeV electron beam at an average current of 60 μA. The three valves are shown in Figure 1. The face of the Valco valve was placed approximately 12 in. from the face of the converter and received an approximate total dose of 1.75 Mrad. The SwageLok and solenoid valves were approximately 14 in. from the face of the converter and received an approximate total dose of 1.13 Mrad.

FIGURE 1 The three valves were tested with flowing pH 1 H_2SO_4 (~0.05 M H_2SO_4) at 40 mL/min in a recycled mode. Top left: SwageLok five-port valve, SS-43ZFS2 (0.062-in. orifice, perfluoroalkoxy polymer [PFA] seat packing). Top right: Peter Paul normally closed solenoid valve, part no. 52X05650GP (24 VDC, 7.0 W, orifice 3/32 in., 175 psi rating). Bottom center: Valco Instrument Co., Inc. 16-port trapping flow-path selector, part no. A6ST16MWE (air actuated, Valcon E rotor).

After 24 h of irradiation, the H_2SO_4 solution turned dark blue. The solution was saved and fresh pH 1 H_2SO_4 was used for the second 24 h period. At the end of the second 24 h period, the H_2SO_4 solution again had turned dark blue. The second solution was also collected. After a period of time, a small amount of black, granular precipitate was observed at the bottom of the containers.

After irradiation, the three valves were leak checked by pressurizing to 10 psig with dry air and monitoring the pressure drop using a pressure transducer. The SwageLok valve is the only valve that held pressure. The Valco valve leaked slowly. Figure 2 shows a comparison of the rotor from the irradiated valve and a new rotor. There was no obvious damage to the irradiated rotor on the left other than a slight change in color. A small amount of dark residue was removed using a Kimwipe from the large channels at the top and bottom of the irradiated rotor.

Figure 3 shows the interior of the irradiated solenoid valve. During irradiation, significant corrosion was observed. The body of the solenoid valve is 316 SS. However, the plunger (piston) that is used to actuate the opening of the valve is made of 430FR stainless steel. The 430FR stainless steel is a ferritic chromium-iron alloy specially developed for soft magnetic components that must operate in corrosive environments (Carpenter 2004). Although 430FR stainless steel has good corrosion resistance to nitric acid, it has restricted use with sulfuric and phosphoric acids (Carpenter 2004). The butyl rubber insert in the end of the plunger retained structural integrity and was not significantly affected by the H_2SO_4. The corrosion of the 430FR plunger resulted in the dissolved material plating out on the surface of the 316SS body. This deposition of material significantly affected the surface near the orifice, impeding formation of a proper seal between the butyl rubber insert and the orifice edge.

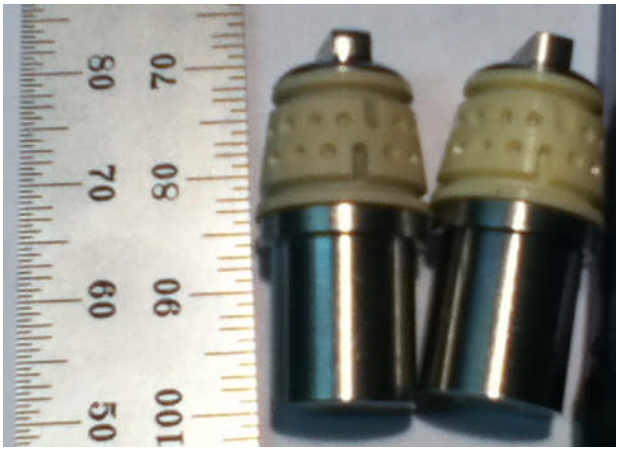

FIGURE 2 Comparison of two Valco rotors for a 16-port trapping flow-path selector valve. (The rotor on the left is from the valve that was irradiated for 48 h at the Argonne Van de Graaff generator. The rotor on the right is brand new.)

FIGURE 3 Interior of the irradiated solenoid valve. Top left: plunger (piston) and outer assembly showing the spring that holds pressure on the plunger to maintain a seal between the rubber insert and the orifice. Top right: magnified view of the tip of the plunger with an arrow showing the interface between the rubber seal and plunger body. Bottom center: body interior showing the orifice (center hole) that is sealed by the rubber seal on the plunger.

There are several types of normally-closed solenoid actuated valves. The Peter Paul valve that was tested is a direct-acting type (Figure 4, top). The solenoid coil (magnetic coil), when energized, draws the plunger up, thus opening the valve and allowing fluid to flow. The process fluid can come into contact with plunger with this type of valve. A spring is used to maintain pressure on the plunger against the orifice in the closed position. In an isolation solenoid valve, a diaphragm is mounted in a fashion that prevents the process fluid from contacting the plunger. A spring is used to maintain pressure on the plunger against the orifice to close the valve. Failure of the diaphragm in the isolation valve would result in the process fluid coming into contact with the plunger material.

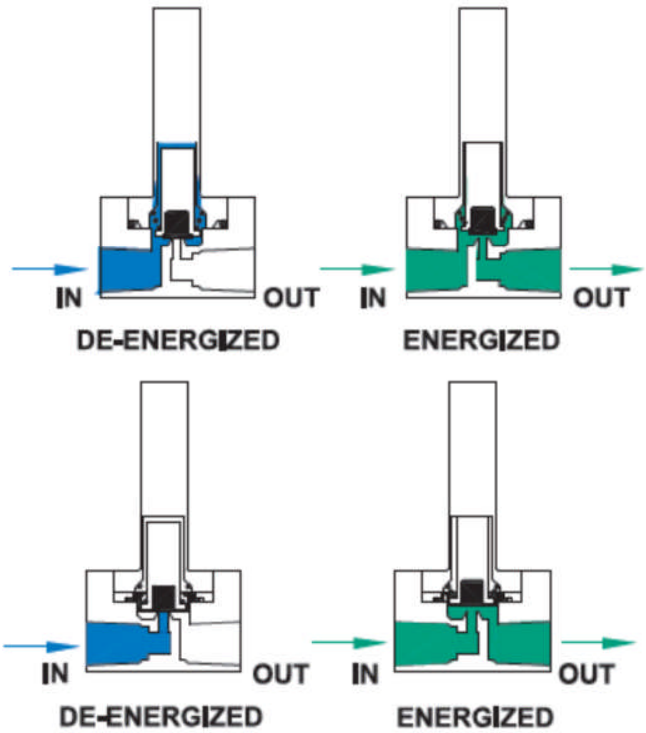

FIGURE 4 Diagram showing interior portions of solenoid valves that come into contact with the process fluid. Direct acting solenoid valve (top) and isolation solenoid valve (bottom).

REFERENCE

Carpenter, May 26, 2004, *Carpenter Stainless Type 430FR Solenoid Quality*. Available at http://cartech.ides.com/datasheet.aspx?i=103&e=63&c=techart. Accessed April 17, 2013.

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