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**In-Service Inspection of the Dissolver
Tank in the Solvent-Refined Coal
Pilot Plant at Wilsonville, Alabama**

S. D. Snyder
B. C. Leslie

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METALS AND CERAMICS DIVISION

IN-SERVICE INSPECTION OF THE DISSOLVER TANK IN THE SOLVENT-
REFINED COAL PILOT PLANT AT WILSONVILLE, ALABAMA

S. D. Snyder and B. C. Leslie

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IN-SERVICE INSPECTION OF THE DISSOLVER TANK IN THE SOLVENT-
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S. D. Snyder and B. C. Leslie

ABSTRACT

A team consisting of a penetrant inspector and a metallographer from Oak Ridge National Laboratory (ORNL) visited the Solvent-Refined Coal (SRC) Pilot Plant at Wilsonville, Alabama, to perform in-service inspection of accessible process surfaces in the top part of the centrifugally cast type 310 stainless steel dissolver tank, its type 347 stainless steel bolting flange, and the flange cover plate. This team and the staff of the plant developed suitable cleaning and surface preparation methods in the field. Visual and dye-penetrant examination located surface cracks, which were also replicated for laboratory metallographic examination. Cracks were found around two nuts that had been welded to the inside of the flange cover plate for attachment of surveillance specimens, and also on the top surface of the flange. No cracks were found on the inside of the flange, the weld attaching it to the type 310 stainless steel dissolver tank, or the accessible portions on the inside wall of the tank.

INTRODUCTION

The Southern Company System and the Edison Electric Institute (EEI) jointly began the pilot plant study of key steps in the solvent-refining process for making low-sulfur and low-ash solid fuel from coal in March 1972. Southern Company Services, Inc. (SCS), represents the Southern Company System and provides management for the project. In April 1973, the Electric Power Research Institute (EPRI) assumed the functions of utility industry project supervision, which were formerly carried out by EEI.

*This work was performed under the Failure Prevention and Analysis in Coal Liquefaction Systems Program sponsored by the Materials and Exploratory Research Branch of Fossil Energy, Department of Energy.

The United States Energy Research and Development Administration (ERDA) joined in the sponsorship of this project for the calendar years 1976 and 1977.

The six-ton-per-day, solvent-refined coal (SRC) pilot plant was designed, constructed, and is operated by Catalytic, Inc., at Wilsonville, Alabama. Operation with coal began in January 1974.

Experience from both the refinery and nuclear reactor industries has shown that under certain conditions stainless steels are highly susceptible to sudden failure due to stress-corrosion cracking. Chlorides and polythionic acid are the principal known inducements for stress-corrosion cracking in coal conversion systems containing large quantities of austenitic stainless steels.¹ These facts, coupled with the identification of stress-corrosion cracking in the dissolver tank of another SRC pilot plant at Fort Lewis, Washington,² prompted the request for inspection service by the management of the SRC pilot plant at Wilsonville.

DESCRIPTION OF EXAMINED PARTS

The parts examined were the accessible surfaces of a type 347 stainless steel flange with its mating cover plate and the cast type 310 stainless steel dissolver tank that had been exposed to the process environment. These parts are shown schematically in Fig. 1. Figure 2 shows the dissolver tank top flange with the cover bolted in place and Fig. 3 shows it with the cover removed. The entire dissolver tank assembly is 7.3 m (24 ft) tall. Figure 4 is a partial view of the pilot plant. The top of the dissolver tank projects above the upper level catwalk where the men are standing.

¹D. A. Canonico et al., *Assessment of Materials Technology of Pressure Vessels and Piping for Coal Conversion Systems*, ORNL-5238, pp. 63-65 (December 1976).

²The Pittsburgh and Midway Coal Mining Co., *Development of a Process for Producing an Ashless, Low-Sulfur Fuel from Coal*, FE-496-T13, Research and Development Report 53, Interim Report 22 for period June 1974-April 1977, pp. 102-105 (May 1977).

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Fig. 1. Cross Section of the Type 347 Stainless Steel Flange Cover Plate and Flange Welded to the Type 310 Stainless Steel Dissolver Tank.

Fig. 2. Dissolver Tank Top Flange with Cover Bolted in Place.

Fig. 3. Dissolver Tank Top Flange with the Flange Cover Removed.
Note the gasket hanging from one of the studs.

Fig. 4. Partial View of the Solvent-Refined Coal Pilot Plant.

PLANT SERVICE CONDITIONS

All components were exposed to the combination of coal, oil, and hydrogen that is fed into the dissolver tank at nominally 415 to 524°C (800–975°F) and 9.6 to 17.2 MPa (1400–2500 psi). Plant operators deliberately vary the operating conditions within these ranges to gain data. During normal operations, the surface below the overflow line is probably exposed to liquid, while above the overflow line the environment is normally gaseous. These parts have been in operation since the plant start-up.

PREPARATION OF SURFACES FOR INSPECTION

The plant management requested surface penetrant examination and field metallography of the upper portion of the dissolver tank to detect and evaluate any stress-corrosion cracking. All surfaces that had been exposed to the process media were covered by tenacious tars and solids,

which required cleaning. Organic solvents such as naphtha and cresol dissolved the tars, but mechanical grinding with a coarse ceramic wheel as shown in Fig. 5 followed by an aqua regia etch in Fig. 6 was necessary for removal of the solids. The surfaces were ground to a smooth finish using decreasing grit sizes of silicon carbide abrasive papers. Then, the surfaces were etched with a solution of 3 parts hydrochloric acid and 2 parts nitric acid (aqua regia) to reveal the grain structure and to remove any grinding debris from the crack system. After etching, surfaces were flushed with water, rinsed with ethyl alcohol, and dried in the ambient environment, 37°C (99°F), ~95% humidity.

Fig. 5. Mechanical Removal of Solids Deposit from the Surface of the Dissolver Tank Flange Cover.

Fig. 6. Etching the Inside Wall of the Dissolver Tank.

The inside wall of the flange and the type 310 stainless steel tank were ground and etched to a region that was well below the 50-mm-diam (2-in.) overflow line about 0.45 m (1.5 ft) into the tank bore.

INSPECTION METHODS

Two inspection methods were employed — visual and dye penetrant. In addition, a plastic film replica was made of any cracks that were found. The penetrant inspection was done with standard red dye and developer. When indications of surface cracking were found, the plastic film replicas were made by applying an acetone-acetate slurry to the cracked surface followed by an overlay of replication tape pressed onto the metal surface. The tapes were returned to ORNL, shadowed by vacuum deposition of aluminum, and examined by conventional microscopy.

No chips of metal were removed for chemical analysis. Therefore, confirmation of the composition of the parts that were examined was not possible.

INSPECTION RESULTS

Cracks on the inside surface of the type 347 stainless steel flange cover plate were evident without penetrant examination, as shown in Fig. 7. The cracks occurred in a 20-mm band adjacent to a fillet weld that attached two nuts to the plate. Surveillance sample holders are normally attached to the nuts. The band of cracks around the nut nearest the thermowell nozzle extended to the intersection of the flange cover plate surface and the wall of the nozzle. These cracks appeared to be very shallow, because no indications extended into the wall of the nozzle. The crack pattern shown at 10 and 100 $\times$ in Fig. 8 suggests a partially intergranular fracture mode. Further work with emphasis on improving resolution might be of value in determining the fracture mode.

Visual and red dye-penetrant examination of the machined top surface of the forged type 347 stainless steel flange between the gasket channel and the inner wall of the tank revealed cracks in four areas. The red dye-penetrant indications from three of these areas and the photomicrographs of the replicas of these cracks are shown in Figs. 9 and 10. The behavior of the dye during penetrant examination coupled with the observation that none of the cracks intersect the inside surface of the tank prompted the tentative conclusion that these cracks were relatively shallow also.

No cracks were found on any portion of the inside wall of the flange or the type 310 stainless steel tank. Photomicrographs of the replicas of the surface at two areas inside the dissolver tank (near the top of the tank) are shown in Fig. 11 at 100 $\times$. The microstructure appears to be that of a fine-grained, equiaxed material in which annealing twins are prominent.

Fig. 7. Cracks Around the Nuts Welded to the Process Side of the Dissolver Flange Cover Plate.
 (c) View through a 3× magnifying glass.

Fig. 8. Photomicrographs Made from Plastic Replicas of the Cracks Shown in Fig. 7 After the Replicas were Shadowed with Aluminum. (a) and (b) Areas around the nuts. 10 $\times$. (c) Grain structure at 100 $\times$. Grain size is ASTM 4.

Fig. 9. A Cracked Area on the Machined Top Surface of the Type 347 Stainless Steel Flange. (a) Arrow shows cracked area as revealed by the red dye penetrant. (b) Replica of the cracks in (a) at 10 $\times$.

Fig. 10. Additional Cracks on the Machined Top Surface of the Type 347 Stainless Steel Flange. (a) Arrows indicate cracks that the red dye penetrant examination revealed. (b) and (c) Replicas of the cracks at 10 $\times$.

Fig. 11. Replicas of the Etched Surface of the Inside Wall of the Dissolver Tank. 100 $\times$.

INTERPRETATION OF INSPECTION RESULTS

Understanding these observations is clouded because of unknowns such as the initial condition of the surfaces that were examined, the metallurgical history of the cracked regions, the levels of stress that were associated with the cracking, and the corrosive agent. However, chlorides, sulfur, and polythionic acids are common corrodants in coal plants. The significant observations and some speculations are:

1. Cracks occurred adjacent to a field weld joining nuts to the type 347 stainless steel flange cover plate. Cracking was not observed at the weld joining the type 347 stainless steel flange to the type 310 stainless steel dissolver tank, nor at the overflow nozzle welds. Thermal cycling during welding and residual stresses are to be expected in the regions where cracking occurred. We speculate that with particular welding methods and parameters on some heats of type 347 stainless steel wrought metal, the aforementioned effects contribute to cracking. Whether these cracks grow into adjacent unaffected base metal is unknown.

2. Cracks occurred on machined surfaces of the flange that were exposed to the process medium. From the location and appearance of the cracks, we speculate that they occurred at regions that were mechanically damaged, or perhaps weld repaired. Only a postmortem investigation or examination of a trepanned plug could substantiate this hypothesis. The growth rates of these cracks cannot be assessed.

3. The microstructure of the type 310 stainless steel dissolver tank is equiaxed, with annealing twins. The tank was centrifugally cast, and some regions of a centrifugally cast pipe might be expected to have a noncolumnar structure. However, only metallographic examination of a cross section of the tank wall can lend understanding to this observation.

4. No cracks were observed on the inside wall of the dissolver tank.

RECOMMENDATIONS

The following recommendations are offered:

1. As a minimum, the cracked regions should be examined periodically to monitor crack growth. Use of ultrasonic techniques should be considered. We recommend that when the SRC pilot plant is scheduled for a downtime of two days or longer, the inside wall of the dissolver unit be examined more thoroughly. An ideal work plan would be to have a metallurgical microscope and a vacuum evaporator at the work site to examine the replicas and repolish and re-replicate any questionable areas. A more stringent recommendation would be to replace all cracked parts.

2. Because cracks occurred at welds and areas suspected of mechanical damage, care should be exercised to avoid unnecessary welding or mechanical damage to regions of pressure boundaries exposed to the process medium.

3. On-line testing of common materials of construction should be initiated to establish the metallurgical conditions, times, temperatures, and stresses that lead to cracking.

4. A metallographic examination of the microstructure of the dissolver tank is recommended to provide useful information on the cast type 310 stainless steel structure, which has apparently resisted cracking.

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