

CASE HISTORY

# Performance of Composite Piping in Concentrated Sulfuric Acid

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*In 1995, a new thermosetting-resin system was introduced to handle concentrated sulfuric acid ( $H_2SO_4$ ) and organic solvents. Previous fiberglass-reinforced plastic technology limited the use of composite piping systems to mild solvents and  $H_2SO_4$  concentrations below 75%. This article shows the results of this thermosetting-resin system after 10 years of continuous service in concentrated  $H_2SO_4$  applications.*

Glass-reinforced thermosetting-resin piping systems have a long history of successful service for handling corrosive fluids in petroleum production, marine, chemical, industrial, and fueling applications. While fiberglass-reinforced plastic (FRP) has been accepted as a material of choice in aqueous and petroleum products, it has not gained industry acceptance in concentrated sulfuric acid ( $H_2SO_4$ ) applications. The phenolic novolac epoxy resin piping system addresses previous limitations with extensive corrosion testing in the laboratory and actual field installations.

The 2-in (51-mm) piping system in this study is composed of a 0.100-in (2.54-mm), eight-layer synthetic veil-reinforced interior corrosion barrier with a synthetic veil and a filament-wound structural wall of 0.111 in (2.84 mm). The manufacturer's maximum temperature rating for 75 to 98%  $H_2SO_4$  is 120 °F (49 °C) with a pressure rating of 150 psig (1.03 MPa).

## Concentrated Sulfuric Acid Installations

Three field installations (Tables 1 through 3) were reviewed for environmental as well as fluid-handling application.

## Procedure

The pipe samples from Applications No. 1 to No. 3 were subjected to a series of physical tests and visual examinations.

## Tests

### ASTM D1599<sup>1</sup>—

#### Short-Time Hydraulic Failure

A section of pipe from each application was placed between mechanical end caps and subjected to hydraulic pressure until the samples showed signs of leakage. The mechanical end caps were unrestrained during the test.

TABLE 1	
Application No. 1	
Geographical location	Upper Midwest USA
Application location	Indoors
In service	May 1997
Samples obtained	December 2008
Total years in service	11.7
System temperatures	0 °F (-18 °C) to 100 °F
Contents	93% H <sub>2</sub> SO <sub>4</sub>
Flow	80 gpm (0.303 m <sup>3</sup> /min)
Pressure	5 psig (0.34 MPa)
Velocity	8.17 ft/s (2.49 m/s)

TABLE 2	
Application No. 2	
Geographical location	Upper Midwest USA
Application location	Indoors
In service	June 1998
Samples obtained	March 2009
Total years in service	10.8
System temperatures	60 °F (16 °C) to 100 °F
Contents	93% H <sub>2</sub> SO <sub>4</sub>
Flow	60 gpm (0.227 m <sup>3</sup> /min)
Pressure	85 psig (0.59 MPa)
Velocity	6.13 ft/s (1.87 m/s)

TABLE 3	
Application No. 3	
Geographical location	Central USA
Application location	Outdoors
In service	October 1998
Samples obtained	August 2009
Total years in service	11.8
System temperatures	-10 °F (-23 °C) to 120 °F
Contents	70 to 93% H <sub>2</sub> SO <sub>4</sub>
Flow	61 gpm (0.231 m <sup>3</sup> /min)
Pressure	60 psig (0.41 MPa)
Velocity	6.23 ft/s (1.91 m/s)

**ASTM D2290<sup>2</sup>—  
Split “D” Tensile Strength**

Five ring samples, ~0.500 in (12.7 mm) wide, were cut from a section of pipe from each application. The rings were placed in a tensile test mechanism and pulled until the rings yielded (Figure 1). The dimensions and load were recorded and the average tensile stress calculated.

**API 15LR<sup>3</sup>—Degree of Cure**

Glass transitions temperatures (T<sub>g</sub>) were obtained by differential scanning calorimeter (DSC) testing for each application from the inside diameter (ID) (corrosion barrier) of the pipe and from the outside diameter (OD) (reinforced structural wall) of the pipe. The glass transition temperature is the approximate temperature at which the resin loses its crystalline structure and becomes amorphous.

**Dimensional Data**

Corrosion barrier thickness (Figure 2) and structural wall readings were taken from pipe wall sections at five locations around the circumference and the average readings were calculated.

**Visual Observations**

The pipe from each application was visually inspected for discoloration, exter-



Split D rings after testing.

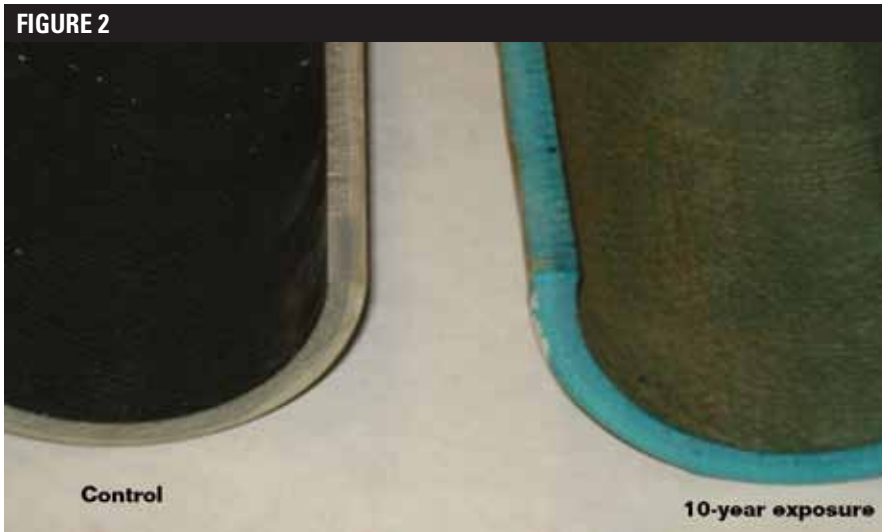
nal ultraviolet damage, and internal liner pitting or deterioration.

**Results**

The test data from the individual applications were compared to an unexposed control sample of pipe manufactured in 1998. Manufacturing tolerances are included for comparison. Pipe sections 24-in (0.61-m) long from each ap-

plication were tested per ASTM D1599 at 3,000 psig (20.7 MPa) with no signs of failure (pipe wall leakage). This was the maximum pressure attainable by the test pump. The piping system tested has a maximum rated pressure of 150 psig (1.03 MPa).

Strength retention represents a baseline number for a properly cured part. The results (Table 4) reflect that 10 years



Liner thickness.

of exposure to concentrated H<sub>2</sub>SO<sub>4</sub> had a negligible effect on the strength of the reinforced wall. Variations from the baseline reflect manufacturing variations and normal aging of epoxy resin systems.

The glass transition temperatures (Table 5) of the corrosion barrier and the structural wall were within manufacturing tolerances. Both the corrosion barrier and the structural wall showed no deterioration of the crystalline structure.

The dull, matte internal surface appearance (Table 6) of Applications No. 1 and No. 2 was similar to a painted surface that had dulled with age (Figure 3). The light green discoloration is the normal epoxy resin reaction to H<sub>2</sub>SO<sub>4</sub> fumes or liquid.

### Conclusions

Exposure to concentrated H<sub>2</sub>SO<sub>4</sub> in temperatures up to 100 °F (38 °C) showed no appreciable deterioration after 10 years of service. A 40% loss in the corrosion barrier was observed in the application exposed to 120 °F, the maximum rated temperature of the piping system, after 10 years of service. This demonstrates that the loss of corrosion barrier thickness accelerates as the maximum allowable temperature is approached. The stability (lack of change) of the Tg (Table 5) and the tensile strength retention (Table 4) of the structural wall indicate that permeation is not occurring.

**TABLE 4**  
**Strength retention of structural wall**

Sample	Tensile Load (lb IN)	Tensile Hoop Stress [psi (N/mm <sup>2</sup> )]	Strength Retention vs. Control (%)
Control	2,088 (9,288)	19,350 (133.4)	—
Application No. 1	2,037 (9,061)	18,134 (125.0)	94
Application No. 2	2,468 (10,978)	21,995 (151.6)	113
Application No. 3	2,414 (10,738)	21,302 (146.9)	110

**TABLE 5**  
**Tg temperatures**

Sample	Corrosion Barrier (°C)	Structural Wall (°C)
Manufacturing tolerances	130 to 145	125 to 140
Control	134	129
Application No. 1	139	125
Application No. 2	130	136
Application No. 3	145	128

**TABLE 6**  
**Average liner thickness/visual observations**

Sample	Liner Thickness [in (mm)]	Visual Observations
Manufacturing tolerances	0.090 to 0.105 (2.29 to 2.67)	Smooth, shiny ID, amber translucent OD
Control	0.103 (2.62)	Smooth, shiny ID, dark amber translucent OD
Application No. 1	0.098 (2.49)	Dull, matte finish on ID; dark amber OD with occasional light green discoloration
Application No. 2	0.100 (2.54)	Dull, matte finish with small amount of pitting on ID; dark amber OD with occasional green discoloration
Application No. 3	0.060 (1.52)	Pronounced orange peel effect on ID, dark amber OD with significant green discoloration

The tensile strength of the structural wall was not affected in either indoor or outdoor installations in 93% H<sub>2</sub>SO<sub>4</sub> applications up to 120 °F service temperature.

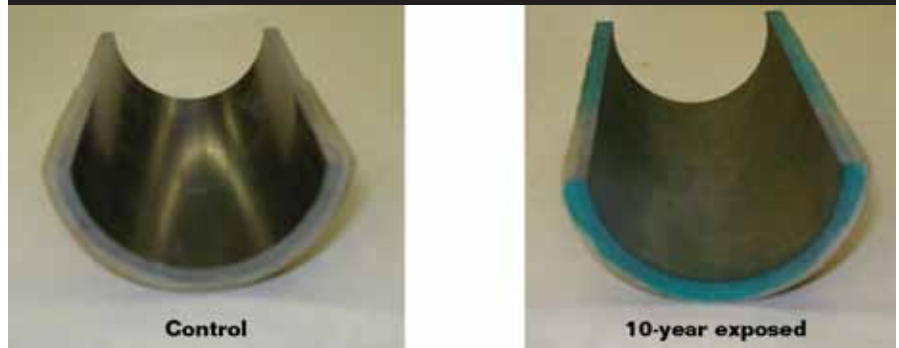
## Acknowledgments

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## References

- 1 ASTM D1599, "Standard Test Method for Short-Time Hydraulic Failure Pressure of Plastic Pipe, Tubing, and Fittings" (West Conshohocken, PA: ASTM International, 2005).

FIGURE 3



Visual.

- 2 ASTM D2290, "Standard Test Method for Apparent Tensile Strength of Ring or Tubular Plastics and Reinforced Plastics by Split Disk Method" (West Conshohocken, PA: ASTM, 2008).
- 3 API 15LR Appendix B, "Method of Test for the Determination of Degree of Cure by Differential Scanning Calorimeter (DSC)" (Washington, DC: API).

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