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 fiberglassreinforced 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.

lass-reinforced thermosettingresin 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 (H₂SO₄) 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% $\rm H_2SO_4$ 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 D1599¹— 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.

MATERIALS SELECTION & DESIGN

TABLE 1		
Application N	lo. 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 ₂ SO ₄	
Flow	80 gpm (0.303 m³/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 ₂ SO ₄			
Flow	60 gpm (0.227 m³/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 ₂ SO ₄		
Flow	61 gpm (0.231 m³/min)		
Pressure	60 psig (0.41 MPa)		
Velocity	6.23 ft/s (1.91 m/s)		

ASTM D2290²— 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 15LR³—Degree of Cure

Glass transitions temperatures (Tg) 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 application 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.

TABLE 4

Strength retention of structural wall				
Sample	Tensile Load (lb [N])	Tensile Hoop Stress [psi (N/mm²)]	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

of exposure to concentrated H_2SO_4 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_2SO_4 fumes or liquid.

Conclusions

Exposure to concentrated H₂SO₄ 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.

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		

FIGURE 3

The tensile strength of the structural wall was not affected in either indoor or outdoor installations in 93% H₂SO₄ applications up to 120 °F service temperature.

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References

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



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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