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INTRODUCTION

The use of Fiber Reinforced Polymer (FRP) for vessels and piping in the chemical processing industry (CPI) started in the 1950's. As experience with the behavior of the material system grew, standards and codes were developed for design and construction that aligned with standards and codes for equipment made with metallic materials. Several features were incorporated to create reliable operation.

One of the design features that evolved to significantly improve reliability of FRP equipment is to incorporate a corrosion-resistant barrier onto the surface of the FRP that is to be exposed to corrosive chemical conditions—usually the inner surface of pipes, tanks or process vessels. The purpose of the corrosion-resistant barrier ("Corrosion Barrier") is to protect the FRP used for structural support (structural FRP) from damage by the operating environment.

When the use of corrosion barriers was introduced, many owner-operators determined that a key to FRP reliability was to properly maintain the corrosion barriers. The principle behind this is to monitor the condition of the corrosion barrier. This approach almost always requires an outage and confined space entry.

This article describes the construction of corrosion-resistant FRP and the practices used for inspecting corrosion barriers, starting from the original visual inspection, to microscopic evaluation of sections through cutouts, and then to an advanced ultrasonic method that yields good correlation to destructive analytic results. Advanced ultrasonic techniques provide additional results that can be directly related to an ASTM standard^[1] that is used globally to provide quantitative performance of FRP in corrosion service.

FRP CONSTRUCTION

Fiber reinforced polymers are used in many corrosive applications because the polymers provide superior corrosion protection to many metal alloys and they also protect the fiber reinforcements that provide structural properties. The surfaces and areas of FRP that will be exposed to corrosive service conditions are covered with a corrosion-resistant barrier.

The corrosion-resistant barrier is normally composed of layers of reinforced thermosetting polymer or a thermoplastic sheet. A thermosetting polymer is a polymer that is applied in liquid form with curing agents added that react with the polymer to form bonds between the polymer chains, known as cross-linking. Examples of thermosetting polymers include epoxy, vinyl ester, and polyester resins. A thermoplastic polymer is a polymer that can be deformed by some combination of heat and stress. Example thermoplastic materials include: polypropylene; polyvinyl chloride; polyethylene; polyvinylidene fluoride; and many others. **Figure 1** shows typical configurations.

Figure 1a shows FRP with a reinforced thermosetting polymer corrosion barrier, consisting of three layers shown as "Veil" and "Chopped Strand Mat", describing types of reinforcement, nominally with a total thickness of 2.4mm. In this case, there is one veil layer and two chopped strand mat layers. Other arrangements may vary the number of layers of each type with corresponding changes to the thickness. Normally a veil layer consists of 90% polymer resin by mass (95% by volume) to offer the highest resistance to chemical attack. The chopped strand mat layers are normally 25 to 30% reinforcement by weight (13 to 16% by volume) and still offer good resistance to chemical attack. The number of layers of veil and chopped strand mat can be adjusted to change the thickness of the corrosion barrier, which also corresponds to the degree of corrosion protection provided. In total, about 85% of the volume (72% of the mass) of a corrosion barrier is the polymer resin. Although it is attached to the structural FRP, the corrosion barrier is not normally considered to contribute any structural properties to the FRP.

Figure 1b shows FRP with a thermoplastic polymer corrosion barrier, or liner. In many cases, the thermoplastic is bonded to the structural FRP, although in some cases the thermoplastic lining is loose. Joints that may exist in the thermoplastic are normally welded by fusion welding. Just as for reinforced thermosetting polymer corrosion barriers, thermoplastics are not considered to contribute to structural properties.

Although most specifications, standards and codes stipulate a corrosion barrier of at least 2.5mm (0.100 inch) thick, some FRP constructions do not include corrosion barriers, while some may include corrosion barriers that are thicker. This reflects variations that may occur because of the preferences of engineers, owners-operators, or manufacturer standards. The thickness of the corrosion barrier and structural layers should be available on drawings for the equipment.

For designers and engineers, determining the construction and materials to be used for a corrosion barrier often starts by using information and analysis that was completed well before the design starts. The polymer used in the corrosion barrier is considered to be the key material—comprising 85% to 100% of the volume in the corrosion barrier and making the FRP leak tight. The small volume of reinforcement present in the corrosion barrier provides only a modest amount of additional strength, and it helps to hold liquid thermosetting resin in place while it cures and it adds toughness for impact resistance.



a. Reinforced Thermosetting Polymer



b. Thermoplastic Sheet

Figure 1. FRP with corrosion-resistant barriers

Choosing the materials for the corrosion barrier can be based on a systematic approach. For thermosetting polymers, the most recognized approach is provided by ASTM Standard Practice C581^[1] where samples that are constructed like a corrosion barrier are exposed to controlled service conditions for up to 12 months. This standard practice allows combinations of polymer and reinforcement materials to be tested. The specimens are evaluated using 3 tests that are completed on specimens drawn from the exposure after: 1 month; 3 months; 6 months; 9 months; and 12 months of exposure. The tests that are completed are:

- 1. changes in hardness of the resin surface;
- 2. changes in weight and thickness of the specimen; and
- 3. changes in flexural modulus of the specimen.

An additional observation is made regarding changes in appearance of the specimen, although no objective criteria are provided within the standard practice. These tests are undertaken

primarily by resin suppliers who then consider the results of this testing, as well as information on similar service applications, to make recommendations of polymers for use. It is important to understand that using ASTM C581 does not result in any recommendations—the standard practice does not draw any conclusions about the performance of specimens, but it does provide some quantitative information that might be related to performance by those skilled in the art. Although the quantitative results from ASTM C581 testing are used to form recommendations for resins to use in FRP construction, they are not used to provide specifications for inspection and repair.

Thermoplastics can be evaluated in a similar method by exposing coupons to the service conditions and taking similar measurements. These tests can be done by owner-operators and polymer suppliers.

It is important to note that any of the results from corrosion testing of thermosetting and thermoplastic polymers are not widely published or available for reference. In addition, the data accumulated in these tests is not used to provide guidance for assessment of corrosion barriers that are in service.

During manufacturing of FRP, the corrosion barrier is built as an integral part of the structure, so measurements of the corrosion barrier are limited to what can be detected from the surface of the corrosion barrier and possibly from examining the edge of a cutout.

HISTORY OF INSPECTION OF CORROSION BARRIERS

Corrosion barriers have been included in most specifications, standards and codes related to construction of chemical-resistant FRP equipment since at least 1969.^[5,6] Experience has taught many operators the importance of assessing FRP for its ability to continue operating or its fitness-for-service. Some owner-operators apply the criteria that damage such as chemical diffusion, chemical attack, or oxidation that extends to the boundary of the corrosion barrier with the structural layers signals the end of service life for the FRP. Although other definitions are available, this definition of the end of service life has served to ensure reliable FRP equipment for many and will be used for convenience in this article. This approach only includes situations where chemicals have penetrated or diffused into the corrosion barrier.

Inspection practices started with visual assessment of the corrosion barrier surface inside FRP containers or pipes. The images in **Figure 2** show surface appearance that may result from a sample of different service conditions. The examples show oxidation, discoloration, scaling, and axial cracking. Note that in all cases, the polymer is opaque and nothing can be seen beneath the surface. These examples come from a wide variety of service conditions and chemical exposures.

In the case of the cracking and the oxidation, it may be possible to estimate the depth of damage without damaging the intact FRP, but the other visible features offer little information about the depth of damage or diffusion of chemicals into the FRP.



Figure 2. Examples of Corrosion Barrier Appearance

There are some resources available to assist with identification of damage.^[78,9,10] Specifications for acceptable levels of different damage types are subjective, leading to poor consensus among inspectors and engineers. In many cases, these inspection guides direct the inspector to standards for new FRP, most of which specifically exclude their use for in-service damage.

For many owner-operators and in some countries, entry into confined spaces is discouraged because of the safety risks to personnel.

Clearly, non-destructive visual inspection of corrosion barrier surfaces has not provided data that can allow prediction of the remaining service life. Inconsistent and subjective assessment of the current condition of corrosion barriers has also left many owner-operators confused.

Some groups have searched for alternative ways to evaluate corrosion barriers to provide measurements that can be used^[11] These other methods often use destructive techniques with microscopic evaluation to measure the presence of chemicals in the corrosion barrier. This work assumes that any chemical diffusion that reaches the boundary of the corrosion barrier and the structural layers defines the "end of life" of the FRP. These methods do not consider the condition of the corrosion barrier, such as provided by ASTM C581 results. Some of the methods proposed include extensive and costly lab testing to provide templates for this analysis-without the lab testing, accurate results are almost impossible to achieve.

Figure 3 shows a specimen of FRP after 17 years in contact with hydrochloric acid. The specimen was made with a corrosion barrier that was 3mm thick. Figure 3a shows the corrosion barrier surface and **Figure 3b** shows the thickness through the







c. Energy dispersive x-ray

Figure 3. Example specimen.

sample-diffusion of hydrochloric acid into the specimen is visible with the green color at the top of the cross-section. Figure 3c shows an electron micrograph of the 3mm corrosion barrier along with Energy Dispersive X-ray (EDX) results for chlorine. Note that chlorine was detected to the full thickness of the thickness shown in the electron micrograph, although Figure 3b clearly shows that visible staining is about 4mm deep. The total thickness of the specimen is 29mm.

Note that Figure 3a does not provide any information regarding the depth of damage or diffusion into the FRP. Figures 3b and 3c provide this information, but at the cost of using a destructive test by removing a cutout from the FRP. Note that these destructive tests require repairs to the FRP and confined space entry. Repair of FRP may interrupt the continuity of the original construction and may create damage prone characteristics in the repaired area if not performed properly.

Although experimental evidence has shown that this destructive approach could work, it is not very practical and will undoubtedly force most work into turnarounds and outages and further extend the time required to determine the current state of FRP assets. Furthermore, unlike the results from ASTM C581, it provides no data or information on whether any actual damage has occurred to the corrosion barrier as a result of the chemical penetration.





a. Ultrasonic reading from outer surface





Figure 5. Some of the samples investigated

Figure 4. Ultrasonic reading from example specimen

Ultrasonic testing has been shown^[13] to provide reliable and valid condition assessment of FRP for its full thickness. The information includes the retained flexural modulus of the material-the same as a key result of ASTM C581 testing. The technique requires post-processing of ultrasonic readings taken using commercially available equipment and following a specific procedure. When normal ultrasonic testing techniques are used, and clear readings are obtained, inspectors often identify "delaminations" within readings because of the appearance of features that look like transverse cracks in metals. In many cases, these "delaminations" signal a situation where the ultrasonic pulse passes through a zone where the sonic velocity of the resin is suddenly different, thus changing the ultrasonic impedance to create a reflection, and not where a defect has appeared. These also appear to be magnified because FRP has a high attenuation factor. These indications can also occur at interfaces of successive lamination stages with no defects present. The sonic velocity of polymers and FRP can be affected by a number of factors, including chemical damage to its molecular structure, the degree of curing, porosity, and others. In general, post-processing analysis is required to identify situations where loss of bonding might be causing the indication and to determine its significance. In some cases, these zones of different sonic velocity exist for the entire lifetime of the FRP and originated in manufacturing.

Figure 4 shows ultrasonic reading information for a reading taken from the outer surface of the specimen from Figure 3. Figure 4a shows a transducer in place with A-scan on the flaw detector. Figure 4b shows the reading after post-processing. The reflection peak at about 14 microseconds is often interpreted as a "delamination." It corresponds to a depth from the inner surface of 5mm. Using non-intrusive inspection techniques^[13] this analysis also allows calculation of the damage to the resin of the corrosion barrier. This is expressed as the change in modulus of the resin that has resulted from the damage. For the example, the corrosion barrier resin has retained 52% of its original flexural stiffness-this is information on actual damage that has occurred to the corrosion barrier and aligns with results from ASTM C581.

This example illustrates a practical ultrasonic method to detect and quantify damage to the corrosion barrier of FRP equipment. The inspection is non-destructive and non-intrusive.

The example above can serve as an illustration, but further evidence is required. For the investigation, we took 30 samples of FRP that have been exposed to a wide variety of service conditions from no exposure to several decades of exposure. The chemical environments included chlorine; chlorine dioxide; hydrochloric acid; sulphuric acid; brines; sulphur dioxide; and others. Figure **5** shows a sample of the sections through the thickness of the samples used. For all photos, the surface with the corrosion barrier is at the top of the image. During the ultrasonic testing, no information regarding service conditions was used to complete the analysis.





Figure 6. Comparison of visible and detected depth



Figure 6 shows a comparison between the visible, measured depth of penetration in the corrosion barrier and the depth detected using ultrasound. **Figure 6** also includes a dotted line that shows where an ideal match would occur. Note that several of the readings found deeper penetration than was visible.

As mentioned above, the ultrasonic data after post-processing can be used to determine the retained flexural stiffness. This allows evaluation of the amount of damage to the corrosion barrier by a subject matter expert. **Figure 7** shows a graph of the retained flexural modulus calculated for the samples in the study. Note that there is no correlation between the visible depth of penetration and the retained modulus of the penetrated material.

Because the analysis provides data on the FRP, the results can be collected into a database including details of the service condition, when available. The data can be used to provide practical, factual data on the performance of polymers and FRP in different corrosion conditions. Using data generated from this analysis should then allow every FRP asset the capability to provide data on the performance of FRP in all service conditions.

A practical non-destructive and non-intrusive ultrasonic technique has been presented for evaluation of the condition of corrosion barriers of FRP equipment. In addition to chemical penetration depth that can be obtained by destructive techniques, the technique also provides information on the condition of the penetrated corrosion barrier. The results can be obtained without requiring details of the FRP materials or the service conditions. The results of this analysis will provide subject matter experts with quantitative data on the performance of FRP assets in service.

For more information on this subject or the author, please email us at <u>inquiries@inspectioneering.com</u>.

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