Smart Materials for Early Corrosion Sensing in Fibre Reinforced Plastics – Final report

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Abstract

Several sensor techniques have been evaluated during the project to investigate the possibility to use them in order to monitor corrosion in glass fibre reinforced plastics (FRP). The project has found very promising techniques as well as less hopeful ones to use for the application. The Lamb wave technique seems to be the most promising one with its easy-to-follow and easy-to-install properties. The technique is also very useful since the sample area in which its function is quite large in comparison with other sensor systems.

There are many ways in which a sensor can be incorporated into an FRP matrix and many different sensors available, which makes it hard to investigate all the possible scenarios. In this report the focus has been on sensor techniques and systems that could be used in the daily life, not just in a laboratory. The goal is to come up with an easily implemented and operated system, that can monitor the critical parameters service life of the FRP components.

Sammanfattning

Under projektets gång har flera sensortekniker utvärderats för att undersöka möjligheten att kunna använda dessa för att övervaka korrosionen i glasfiberförstärkt plast. Vissa av teknikerna verkar mycket lovande medan andra kräver mer arbete för att kunna utnyttjas. Lamb-våg-tekniken anses vara den mest lovande av de tekniker som har testats under detta projekt, detta på grund av sin enkelhet att använda och dess förmåga att kunna avläsa ett relativt stort område i jämförelse med andra sensorer.

Det finns många olika sensorer tillgängliga och därmed också sätt på vilka dessa kan inkorporeras inuti ett GAP-laminat, detta har gjort att projektet behövt begränsa sig och alla tänkbara scenarion har inte kunnat testats. I denna rapport läggs fokus på sensortekniker som skulle kunna användas i vardagen, inte endast på laboratorium. Målet är att kunna övervaka reell utrustning ute i fält/industrin och detta ska kunna genomföras av arbetarna själva på ett enkelt vis.

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1 Objective

The aim for this project is to study how sensors available on the market can be incorporated and used as corrosion detectors in constructions of fiber reinforced plastics (FRP). The results will support and help users and suppliers of process equipment to detect corrosion as it happens and prevent corrosion problems in time. Corrosion monitoring enables resources and energy savings due to less unplanned and shorter planned shutdowns, a better way of maintaining the assets. Today, the main way of detecting corrosion problems in FRP is during downtime periods and often requires destructive testing of material cut-outs. A corrosion sensor integrated into polymeric materials will allow early detection of corrosion and thus reduced precautionary maintenance. It also opens for digitalization of the plant maintenance which will generate higher value for the end-user than an inspection would have done.

The overall objective of the project is to create smart fiber reinforced plastics for corrosive environments by evaluating inexpensive sensors already available on the market for corrosion detection. The wireless monitoring will increase the safety of the workers and provide crucial information about the status of the FRP structure.

2 Introduction

2.1 Condition monitoring

Condition monitoring is made by measuring the same equipment and/or system continuously or at regular intervals to get indications about possible changes in properties. The idea behind this project is that the material for the construction will incorporate this monitoring function. For a successful condition monitoring there are a number of important parameters to consider. First of all, it needs to monitor a relevant change in property, i.e. it might be irrelevant to measure water up-take if it is severe loss of material by erosion that is the service life limitation. The system also needs to be stable over time so that the sensor does not drift or stops functioning. In corrosive environments this can be quite challenging to achieve. The sensor should also not negatively affect the service life of the material. The system should also be sensitive enough to respond to relevant changes in properties without being overly sensitive. The latter might take time to fine-tune for a system.

2.2 Fibre reinforced plastic (FRP)

FRP is built up by mainly three components; resin, glass fibres and additives. The glass fibres give the mechanical strength to the FRP-construction and the resin holds the composite together and gives corrosion resistance. The glass fibres are dependent on a good adhesion with the resin matrix in order to acquire strength in all directions of the composite. It is of great importance to have a composite where the resin and glass fibres allows a good interface, not only to enable load transfer but also to avoid penetration and corrosion along the glass fibres. Depending on the type and amount of each of the components used, the final product can be designed in many ways to make it suitable in various applications.

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2.2.1 Glass fibres

In order to design the mechanical properties of the material, different glass fibre structures are used. Some examples are shown in Figure 1, there are also other kinds of glass fibre structures on the market. Chopped strand mat (CSM), woven roving and roving are used for FRP materials for the processing industry. The glass fibres in the CSM mat has no general direction, while in the woven roving mat, the glass fibres have two distinct directions, this results in different properties of the final laminate. There are also different types of glass available on the market, the dominating ones being; E-glass, C-glass and ECR-glass. E in E-glass stands for electrically insulating. C in C-glass is for chemical resistance. ECR-glass has properties somewhere in between E-glass and C-glass. Depending on the application in which the FRP shall be used the manufacturing technique can be either by hand lay-up or by machine. With a machine it is possible to exactly control the angles between the fibres and in that way design the mechanical properties in precisely the right way for the application, see Figure 2. When making a laminate by hand it is not possible to control the structure in the same way as with machines, it is more of a craft. [1]



Figure 1. Different structures of glass fibres. Woven roving (A), Chopped Strand Mat, CSM (B), synthetic woven polyester (C) and roving (D). [1]



Figure 2. Manufacturing of tank in FRP made by a machine. [1]

2.2.2 Resins

The resin used for FRP in process equipment are often of vinyl ester (epoxy) type but can also be polyester based. Vinyl esters are being used in process equipment for their high corrosionand temperature resistance. Styrene is being used as a reactive solvent in these materials to reduce the viscosity and to crosslink the resin during the curing process. There are different methods that can be used for the curing of the resins. A hardener (peroxides are commonly used) is added to the resin and a chemical curing process starts, often an increased temperature is required to complete the curing. To design the material even more, different additives can be used. [1]

2.2.3 Build-up of laminate

Figure 3 shows a schematic view of a general laminate build up for FRP used within processing applications. Closest to the chemical there is a resin rich corrosion barrier (about 2.5 to 5 mm thick) with a low content of glass (in the top of the figure). The glass fibre in this layer is usually a surface veil and chopped strand mats (CSM), i.e. it has no general fibre orientation. Outside this is the structural bearing layer with much higher glass content, usually wound fibre and/or woven fibre mats with a preferential fibre direction (in the bottom of the figure). [1]



Figure 3. Schematic picture of an FRP laminate, at the top the corrosion barrier (red arrow) and at the bottom the structural bearing layer. [1]

Figure 4 shows a cross section of a real FRP laminate, here the corrosion barrier layer and the structural bearing layer clearly can be seen. The corrosion barrier is not load bearing and corrosion of this layer can be allowed. However, no corrosion can be permitted in the structural support layer since that can result in decreased mechanical properties for the construction.



2000 µm

Figure 4. Cross section of a typical GRP laminate for corrosive environments showing the corrosion barrier and the structural laminate.

2.2.4 Corrosion of FRP

Different chemicals and environments can attack the FRP material and start corrosion processes. Common chemicals that can cause corrosion in FRP in the process industry are acids, bases and even water. Design of the FRP material, the concentration of chemicals, temperature, pressure and time are all parameters that will have an influence on the corrosion processes. Figure 5 shows a schematic picture of the different corrosion mechanisms that can occur in polymer matrix composites as a result of moisture. There are several ways water and other liquids can attack FRP and influence its properties. Depending on the properties of the solvent some of them can, e.g. diffuse into the matrix because of osmosis, liquid absorption in the interface between glass fibres and matrix and swelling of the resin matrix. A typical effect on FRP when water diffuses into the material is that the E-modulus decrease, as a result of debonding of the glass fibres from the resin matrix when water is absorbed in the interface between fibre and matrix. This is something that must be taken into account when designing applications in FRP that has the demand on take a certain load.



Figure 5. Schematic picture of the effect moisture can have on FRP.

2.2.4.1 General corrosion

Corrosion of plastic materials can be divided into different types of corrosion mechanisms. General corrosion, i.e. corrosion that can be measured in mm/year, can be the result of exposure in acids or bases. If the environment is constant it is possible to predict this type of corrosion since the rate often is proportional to the square root of time, highest in the beginning and slower with time. Experiments performed on FRP material in bases at different temperatures showed that the corrosion rate increased with higher temperature and that different kinds of FRP materials was affected in different ways. This shows that the material composition is of utter importance for the corrosion properties. [2]

2.2.4.2 Local corrosion

Local corrosion is just what it sounds like, corrosion on local places on the material. At these places the corrosion rate is higher compared to other places on the material. Examples of places where local corrosion can appear are areas with defects such as small damages or fibres that are not embedded inside the resin matrix.

2.2.4.3 The effect of water on FRP

Water can have a big influence on FRP materials. When water diffuse into the material the Emodulus will decrease as can be seen in Figure 6. [3] Depending on which type of resin that is used the diffusion of water will vary between materials. Water molecules can also diffuse into the resin via osmosis to be dissolved and eventually forming blisters. If the pressure inside the blisters gets too high, they can crack and cause failure of the structure. Delaminations of layers inside the composite is another defect that can be seen in FRP and in worst case scenario they can cause failure of the structure. [1]



Figure 6. E-modulus and strain as a function of time for an FRP material exposed in water. [3]

2.2.4.4 Stress corrosion

A material under the simultaneous action of stress and chemical exposure can be subjected to stress corrosion. The most common case is when the material is exposed to some sort of acid. Stress corrosion can only happen if the acid has direct contact with the glass fibres and because of this it is not good to have free fibres or damaged laminates where the acid can penetrate. Stress corrosion failures are often very rapid and therefore often very serious. E-glass is more sensitive towards stress corrosion compared to the other glass types. [1] Failure due to stress corrosion cracking have been reported from e.g. tanks and pipes for hydrochloric and sulphuric acid and chlorine dioxide.

2.2.4.5 Erosion corrosion

Erosion corrosion is when the erosion of the material is being accelerated by corrosion. This type of corrosion can happen in applications where it is high flow rate in a pipe for example. Erosion corrosion influences the thickness of the corrosion barrier and this uncovers the fibres which makes it easier for other kind of corrosion mechanisms to occur. Just like all other kinds of corrosion, erosion corrosion is not permitted in the structural laminate. [1]

2.2.4.6 A real example of corrosion of an FRP material from the industry

An example of a common corrosion problem for the processing industry is attack of HCl on FRP, see Figure 7. The picture shows a cross section of a laminate that has corroded due to penetration of hydrochloric acid. Here the corrosion can be seen as a green discoloration in the laminate (upper part of the cross section) and it is clear that the acid has penetrated about 2/3 of the thickness of the corrosion barrier. When a cobalt accelerator is used in the material the laminate is colored green by the hydrochloric acid. But if cobalt had not been used the penetration of acid cannot be seen. As said earlier corrosion of the corrosion layer can be permitted but as soon as it reaches the structural bearing layer it cannot be tolerated since this will lead to changes of the mechanical properties of the laminate structure. Because of this it is very important to be able to detect the rate of the corrosion into the laminate. One example of

a corrosion mechanism that can be found after attack of HCl is micro delaminations in the corrosion barrier. Today the only safe method to do this is to take out a physical sample from the process equipment and polish a cross section of it as in Figure 7. This is often not easily done and requires a shout down of the system in order to take out a sample for analysis.



Figure 7. Hydrochloric acid penetration, seen as a green discoloration, in the corrosion barrier.

2.3 Smart FRP

The project idea is to use inexpensive sensors that can be read remotely to detect diffusion and penetration of a corrosive chemical before it reaches the structural bearing layer. Creating a smart sensor material for detection of corrosion would be of great significance for diverse industrial areas such as chlorine production, pulp and paper mills, and water treatment, only to mention a few. If it would be possible to use this kind of sensors it would be much easier to discover potentially very dangerous changes in time in the FRP properties due to corrosion.

3 Approaches to reach smart FRP

A number of different sensor techniques were evaluated with the help of several experimental setups during the project. Each of them will be described in detail in this chapter.

3.1 Fibre Bragg Grating (FBG) Sensors

3.1.1 Background

Together with Acreo Swedish ICT (RISE), initial testing was conducted using standard fibre optical sensors for telecom and fibre bragg grating (FBG) sensors. The reasons for using these types of sensors are due to the sensors being small, lightweight, compatible with harsh environments (high temperature and pressure), multiplexing capabilities along a single fibre and because they are currently used for health monitoring of civil structures made of concrete and/or FRP.

There are different types of optical fibre sensors, as can be seen in Figure 8. A point sensor only has a single sensor location while a multiplexed sensor has multiple sensor locations along the fibre. Distributed sensors have a continuous sensor along the whole length of the fibre.



Figure 8. Different types of optical fibres.

An FBG-sensor (Fibre Bragg Grating) has a periodic or almost periodic structure consisting of a variation of e.g. the refractive index, along the length of the waveguide. A schematic picture of an FBG-sensor can be seen in Figure 9 and Figure 10. The refractive index profile will give a change in the spectral response, so that the transmitted and reflected light will give a fingerprint of the specific refractive index profile of the optical fibre.



Figure 9. Schematic picture of a Fibre Bragg Grating (FBG) sensor.



Figure 10. Schematic picture showing how the spectral response is affected by the core refractive index.

By designing an FBG optical fibre which has several local gratings as independent spectral filters a spectral encoding can be done by recording shifts of the wavelengths. A certain shift in wavelength can then be correlated to a deformation of the optical fibre such as strain or compression. As a rule of thumbs for the sensors used in this project, a variation in temperature of 1 °C causes a wavelength shift of 10 pm and a strain of 10 $\mu\epsilon$ causes a wavelength shift of 12 pm.

In this project, FBG-sensors were incorporated in FRP samples and exposed to water and heat at 70 °C. The idea of the experiment was to measure the strain on the glass fibres. During the experiment the FBG-sensors were connected to a computer for continuous monitoring. The strain on the glass fibres, coming from thermal expansion, was successfully measured.

3.1.2 Experimental

As a start, two types of FBG sensors were laminated into FRP samples, see Figure 11, one of the FBG sensor being a polyimide coated fibre and one an acrylate coated fibre.



Figure 11. Schematic illustration of the FBG sensor in the middle (red) of four CSM layers.

The FRP laminates cured and, after curing, they showed a shrinkage that could be recorded by the sensors, therefore the experiment can be considered promising. The laminate was tested in an oven at 70 $^{\circ}$ C with single sided exposure to water, see. The results of this experiment showed that the wavelength increased with time, which corresponds to the thermal expansion of the laminates. These results can be seen in Figure 12.



Figure 12. The experiment setup for the single sided exposure to water at 70 °C.



Figure 13. Results from the single sided exposure of GFRP laminate to water at 70 °C.

The strain in the GFRP sample when putting it into the frame for the single sided exposure could be seen with the sensors, as well as the thermal expansion while increasing the temperature from 20 $^{\circ}$ C up to 70 $^{\circ}$ C. The wavelength shifted with time of the exposure and this was due to the thermal expansion of the FRP.

3.2 Resistance measurements

3.2.1 Background

Continuous monitoring of the resistance of thin metal films embedded in an FRP sample have been carried out. The FRP sample was exposed one-sided to HCl. The idea was that when the acid diffuses through the FRP sample and reaches the thin metal films, they will start to corrode and change their resistance. This could then be used as an indicator of how far the acid has diffused through the FRP sample.

3.2.2 Experimental

Four different sized thin metal films were incorporated into FRP laminate, as can be seen in Figure 14. Three of the thin metal films were successfully coupled to cables with different colors to be able to measure the resistance, see Figure 15 and Figure 15.



Figure 14. The FRP sample during manufacturing, the position of the thin metal films can be seen.



Figure 15. The finished sample with cables coupled to the thin metal films inside.

The FRP sample was exposed on one side to concentrated HCl at 50 $^{\circ}$ C in an oven. During the whole exposure time of 90 days, the resistance of the incorporated metal films was measured. The results are illustrated in Figure 16, the resistance versus exposure time.



Figure 16. Resistance versus time of exposure for the three thin metal films incorporated in the FRP laminate.

As can be seen the resistance did not change over the exposure time at any of the thin metal films. The reasons for this can be several but two possible causes are: too short exposure time so that the acid didn't diffuse all the way to the metal films and/or lack of oxygen inside the FRP laminate to cause metallic corrosion/change in conductivity.

Microscopic images were taken on cross sections of the laminate to see how long the acid had penetrated, see Figure 17. It is very hard to tell if the acid diffusion reached all the way to the thin metal films or not. Further investigations are needed in order to evaluate this method more thoroughly.



Figure 17. Microscopic picture over the cross section of the laminate with incorporated thin metal films after 90 days exposure in HCl.

3.3 Radio Frequency Identification (RFID)

3.3.1 InviSense humidity sensor

To investigate the possibility to use RFID-technique to monitor corrosion in FRP sensors from InviSense were assessed. The InviSense-sensor (Figure Invi-1) is a new product on the market

that enables wireless relative humidity (RH) measurement by using a passive sensor and detector device. The sensor has been developed for the building industry to monitor water damages. The sensors can e.g. be installed behind or inside the walls at crucial positions, during construction of a bathroom. When the construction work is completed the sensors can be read through the wall using the for-the-purpose-developed InviSense scanner to acquire a relative humidity value at the sensors position. In this way moisture damages that occurs inside or behind the wall can be detected in a convenient way.

The sensor is passive, which means that it only measures and communicate when a current is induced by the InviSense scanner. The sensor works when an electric field induce a current in the cupper coil of the sensor (Figure Invi-1). In the middle part of the sensor a capacitor with a moisture sensitive polymer as dielectric is placed between the two plates. Depending on the amount of adsorb water in the polymer the resonance frequency of the capacitor is shifted. The resonance frequency of the capacitor shows a linear relationship against the RH and thus the RH can be found by identifying the resonance frequency (Figure Invi-2c). When scanning with the scanner a frequency sweep is performed until the resonance frequency is found. [4]



Figure Invi-1a) InviSense moisture sensor b) layer buildup of the sensor c) the relative humidity of the surrounding shows a linear relationship with the frequency of the dielectric polymer. [4]

Thanks to the design, the InviSense sensor is only 0.5 mm thick, 6 cm wide and 7 cm long. Considering the flat dimensions and being wireless the sensor makes a good candidate to install in FRP to enable a smart FRP material. To be able to monitor the water content in FRP can be very valuable for condition monitoring.

A drawback when introducing a sensor can be that it works as a defect, but in the case of the InviSense-sensor it should not be an issue to added it, as long as the application mainly have a pressure that is not lower on the inside than the outside of the FRP-construction.

3.3.2 Experimental

The sensors were embedded in Derakane 470HT, a resin commonly used within processing industry with tolerance to high temperatures. Initially the sensors were embedded in a single step to the resin, but it turned out that during the curing of the resin the sensor stopped working. It is unclear what was the reason for the failure, if it was related to a sensitivity of the sensor to the resin or a reduced possibility for the condenser to vibrate due to an expansion of the resin after curing, or any other reason.

In order to circumvent this issue, the test samples were made in two steps. First a foundation was laminated and then the sensors was placed on that. On top of the sensor a plastic film was then added in order to limit the direct exposure of the sensor to the resin used for the continued lamination on top of the sensor. This worked out and it made it possible to acquire an RH-value from the sensor when embedded in the resin. Two different approached to produce the laminates were performed, either with pure resin on top of the sensor to create a clear cast or with glass fibres (chopped strand mat - CSM) next to the sensor. A drawback with the addition of a plastic film on top of the sensor was that the interpretation and predictability of the experiments more challenging.

Sample	Kind of exposure	Media	Temperature (°C)	Comments
1	Single side	H ₂ O (l)	70	Only diffusion through clear cast
2	Single side	HCl (g)	70	Only diffusion through clear cast
3	Single side	H ₂ O (l)	70	Only diffusion through clear cast
4	Double side	H ₂ O (l)	70	Exposed glass fibres on the edges,
5	Double side	$\begin{array}{c} Air + H_2O \ (g), up \\ to \ 70\% \ RH \end{array}$	70-185	Exposed glass fibres on the edges,

Table Invi-1. The five sensors embedded and exposed

Three samples were single side exposed in a cup (Figure Invi-2), two with distilled water and one in 37wt.% HCl. These samples had air inclusions and penetrating channels on the backside from the fabrication which made them more suitable for single sided exposure. The two other samples where more uniform (Figure Invi-3), but they had CSM on top of the sensor and no edge sealing. This facilitate penetration of water molecules along the fibres, increasing both the rate and the amount of adsorbed water. One of two samples was totally immersed in distilled water at 70 °C and the other sample was inserted in the flue gas stack test bed. In the test bed the temperature was cycled between 70 °C and 185 °C and during running at 70 °C water mist was injected via a spray nozzle (see Section 3.5.2).

All measurements were performed at 70 $^{\circ}$ C, i.e. the sensors were scanned when just taken out from the furnace.



Figure 18-2. Sample 1 (a) before exposure and (b) sample 1 and 3 as mounted for single sided exposure in water at 70 °C. The same setup was used for Sample 3 but then turned upside down to expose the FRP-laminate for HCL gas.



Figure Invi-3. Sample 4 with an InviSense-sensor incorporated in an FRP laminate. To the left (a) the sample before double sided exposure in distilled water and to the right (b) the same sample immersed in distilled water. Sample 5 looked the same as sample 4.

3.3.3 Results

Figure Invi-4 shows the relative humidity of sample 1-4 up to 100 days of exposure. Sample 1-3 had before curing an RH around 25%, once embedded and cured the sensors all dropped to a relative humidity of 10%. It seems as during curing water is removed from the sensor to the resin. The same behavior was not seen for sample 4 and 5 that had glass fibres (CSM) on top (and a different plastic cover), during curing they did not go down in RH. Probably an effect from being more protected from the resin or larger air voids in the vicinity to the sensor. Once exposed it can be seen that the fully immersed sample (sample 4) rapidly increases in RH but does not reach 100% until after 30 days. After 80 days the sample was removed from the water

and put into a furnace for drying. Also, at 70 °C. When doing this the RH decreased down to 10 % RH in roughly a week. It is clear that the sensors work in reversible mode, also when embedded in resin. In Figure Invi-5 sample 4 is displayed before and after exposure. When exposed to water the CSM on top of the sensor get whitish and less transparent, this is an effect that is not reversible.

Sample 1, starting from 10% RH jumps to 30 % in only 5 days, after that it shows a slow increase in the RH, reaching 43% after 101 days, probably related to the diffusion of water through the resin. The same trend can be seen for sample 3 but with a more modest increase in the beginning followed by a long-term slow increase, reaching an RH of 36% after 101 days. The step wise increase in the beginning if sample 1 is unexpected, since the diffusion process should not be that rapid.

No change in signal was detected for the hydrochloric acid. The aim of this experiment was that diffusion of HCl into the sensor would lead to a failure of the sensor and a subsequent loss in response when using the detector. Unfortunately, this was not reached during the exposed time. Sample 1 and 2 after exposure can be seen in Figure Invi-5.



Figure 19. Relative humidity plotted against time of exposure.



Figure Invi-5. InviSense-sensor embedded in FRP, (a) before and (b) after dubbel sided exposure in distilled water.

Figure Invi-6a-c shows sample 1 and 2 after exposure. Sample 1 shows no visual difference in the material after exposure, this since there were no glass fibres on top of the sensor. For sample 2, exposed to HCl a brown discoloration can be seen (exposed surface in Figure Invi-6b and backside in c), which is a result of a chemical attack of the HCl on the resin.



Figure 20a-c. Sample 1 and 2 after single side exposure to (a) water and (b) HCl where the backside of the HCl is seen in (c).

Sample 5 was added to the in-house flue gas stack (see Section 3.5.2). This sensor had an initial RH value of 26 but decreased to 10 % when it was exposed to cyclic heating up to 185 °C. Since the cyclic heating was ongoing continuously no change was observed until a long period of water injection at 70 °C was performed. Then it raised to 17 % after a couple of days. The sensor after exposure can be seen in Figure Invi-7a. Here the sensor is hardly visible due to discoloration of the resin when exposed to elevated temperatures. Also, the CSM is influenced by the elevated temperatures.

After exposure it can be seen for all transparent samples that the cupper has partially corroded and turned green. In Figure Invi-7b the backside of sample 3 is shown, where large areas in connection with the resin turns green. It is unclear why the cupper corrodes when in contact

with the resin. It might be a combination of contact with the resin and elevated temperatures. Usually this green discoloration is associated with sulfuric corrosion compounds of the copper.



Figure Invi-7a. Sample 5 after exposure in the flue gas stack and (b) the backside of sample 3 after exposure, where it can be that the cupper has turned green due to corrosion.

3.4 Electrical impedance spectroscopy (EIS) to study water and HCI diffusion

3.4.1 Background

Electrical impedance spectroscopy (EIS) was used to investigate its potential as a nondestructive testing method for diffusion and conduction of ions in FRP and clear casts. EIS is a well-established method when it comes to characterizing properties of thin organic coatings on metals, such as paint, and is used in e.g. standard ISO 16773 [ISO 16773]. While it is commonly used for thin coatings it has gain limited attention when it comes to characterizing and understand transport properties of FRP and its clear casts. [5] A clear cast is when no fibres are used, only resin. Thus, only the properties of the cured resin are studied when looking at clear cast. Since penetration of water may lead to a loss in mechanical properties for FRPconstruction [3] and an accelerated corrosion it is of importance to enable monitoring of water levels over time. Thanks to the large difference in dielectric response between polymers (in general a dielectric constant < 10) [5] and water (80 at 20 °C) [6] EIS may turn out to be such a method that enables a long time monitoring of water uptake in FRP.

EIS is performed by measuring the voltage-current response of the polymer when applying a frequency sweep. The response is depending on the state of polarity and mobility of the medium in between the two measuring electrodes. While polymers have a low dielectric constant ($\varepsilon_{polymer}$), i.e. low ability to align to the changing electrical field, a sharp change in the response signal can be seen when water and other small molecules with high dielectric constant have diffused into a polymer-based system ($\varepsilon_{polymer+solute}$). A schematic picture of the circuit can be seen in Figure 21.



Figure 21. Schematic picture showing the circuit of the electrical impedance measurements on a polymer sample.

3.4.2 Experimental

EIS experiments were performed on test coupons, in accordance to Table 1. Sample composition and parameters for the experimental set-up, that were fully immersed in water and HCl 37 wt.% at 50 °C. Laminates with one or two carbon fibers strands, acting as electrodes, were single sided exposed for water at 80 °C.

Sample	Composition	Exposure media	Temperature (°C)
1A	Atlac430	HCl, H ₂ 0 – double sided	50
1B	Atlac590	HCl, H ₂ 0 – double sided	50
2A	Atlac430 with GF	HCl, H ₂ 0 – double sided	50
2B	Atlac590 with GF	HCl, H ₂ 0 – double sided	50
3	Derakane411-350 with GF + single CF	H ₂ 0 – single sided	80
4	Derakane411-350 with GF + double CF	H ₂ 0 – single sided	80

Table 1. Sample composition and parameters for the experimental set-up

Atlac 430 (Aliancys) is an epoxy bisphenol A vinyl ester and is commonly used in processing industry with high corrosion resistance to a wide range of acids, alkali and bleach (as described in product data sheet). Atlac 590 is an epoxy novolac based vinyl ester with similar properties as Atlac 430 and is used in processing industry (as described in product data sheet). Atlac 590 has a greater resistance to higher temperatures compared to Atlac 430. Derakane 411-350 is an epoxy bisphenol A vinyl ester produced by Derakane and is commonly used under the same conditions as Atlac 430.

In Figure 22 the single and double strand CF samples can be seen. The inclusion of conductive CF enables the possibility to simplify continuous or long-term measurement at a single spot over time. CF can be introduced in common glass fiber plastics without compromising other properties, it is thus a feasible way to include a permanent sensor.



Figure 22. To the left single strand CF, to the right double strand CF samples.

In Figure 23 the single sided exposure of sample 4 can be seen.



Figure 23. Single sided exposure of sample 4.

Measurements were performed on an Autolab Potentiostat measuring in the frequency range of 1 MHz to 1 Hz with voltage amplitude of 50 and 150 mV, which works fine with such resistant material as FRP. A faraday box was used to avoid disturbance and the measurements were performed at 20 °C immediately after the sample was picked up from exposure medium.

3.4.3 Results

Figure 24 shows the Nyqvist plot of Sample 1A (no GF) exposed for H_2O and HCl up to 27 days.



Figure 24a and b. Nyqvist plot of sample 1A exposed to (a) water and (b) hydrochloric acid.

In Figure 25, when the clear cast is exposed to water a deviation from straight vertical line, and purely capacitive behavior, can be detected after 648 h (27 days). This indicate ionic conduction which may seem unexpected for the H_2O -exposed sample but may be explained by dissolving of salts from the glass fibres. A process known to be the reason for blistering, where dissolved salts in the FRP creates an osmotic pressure and a driving force for increase water diffusion into the material. For the same sample, but exposed to HCl, the loss of purely capacitive behavior was seen already after ca 100 hours. After this the ionic conduction increased steadily. The same behavior was also seen for sample 2A but not with as high conduction.



Figure 25a and b. Nyqvist plot of sample 2A exposed to (a) water and (b) hydrochloric acid.

When GF was embedded in the resin it could be seen that there was a slow increase in the proportion of ionic conductivity in water. For the HCl exposure on the other hand it was already after the first measurement (50 h) possible to see a marked ion conductivity, detected as a formation of an arc. This arc becomes smaller for each measurement, indicating an increased ionic conductivity over time. It can be expected that the conductivity takes place along the

fibre/matrix-interface, where an increased conductivity can be suspected related to degradation of interface bonding, increasing the volume dissolved ions and thus ionic conductivity.



Figure 26a and b. Nyqvist plot of sample 2B exposed to (a) water and (b) hydrochloric acid.

The same behavior was seen for Sample 2B but with a tendency towards more susceptible to water diffusion. This is can be related to the properties of the interface, where the surface treatment of the used glass fibres might give better interface for the Atlac 430 than Atlac 590.

Sample 3 and 4 were exposed in water at 80 °C for 3.5 months. Sample 3 had one carbon strand imbedded in the sample as measurement electrode, and a steel plate was used as a second electrode. From the EIS a weak ionic conduction could be detected already after 22 h, see Figure 24. At the same time an increase in the dielectric constant of the exposed resin in between the carbon fibre strand and the steel plate was seen. This shows that it is possible to detect the water that diffuse into the material with use of embedded carbon fibre as electrode material.



Figure 27. Nyqvist plot (a) of Sample 3 and the calculated dielectric constant as a function of time exposed to water.

The measurements showed that the response went from purely capacitive with a high resistivity at the beginning to become more and more conductive with time, i.e. diffusion of water and dissolving of ions increased over time in all cases. It was clear that the glass fibres enhanced the diffusion compared to the clear cast (without fibres) and that HCl, compared to pure water, more readily attacked and penetrated the samples since the samples exposed to HCl showed an early increase in conductivity.

For Sample 4 it was not possible to detect any ionic conduction even after 5 months of exposure in water at 80 °C. A change in the dielectric constant from 3 to 3.5 during exposure could though be detected, this gives an indication that water to limited extent has diffused in between the fibers.

Considering the greater response when exposing to HCl it may turn out that embedding of carbon fiber in the FRP may be a possible technique to study early corrosion. Thanks to a more powerful response in dielectric changes when using HCl as solute, it would be a good idea to investigate the response of carbon fibre test coupons when exposed to HCl. It may very well turn out that it can be a very powerful tool to monitor HCl diffusion in an FRP laminate.

3.5 Structural Health Online Monitoring of FRP Equipment using a Lamb wave technique

3.5.1 Background

Lamb waves are acoustic waves that can exist in thin plate-like structures with parallel free boundaries, such as pipes. Lamb wave can travel over long distances, even in materials such as FRP, but are highly susceptible to interference along the propagation path. The properties of the lamb waves, long propagation and high susceptibility, makes it suitable for non-destructive testing (NDT), which was suggested already in 1961. [7] Since the lamb wave propagates in the whole thickness of the plate it is possible to detect both internal and external damages in the composite. Lamb waves have several properties for NDT: 1) large structures can be inspected without removing coatings or insulations 2) the entire cross-section of the pipe can be inspected and 3) low energy consumption to generate waves [8]. A simulation of the propagation and the change in response signal due to interference with a damage can be seen in Figure L1a-c.



Figure L1a. Interaction of defects with lamb waves in complex geometries. b) lamb waves on defect areas where the blue lines represent the baseline signal and the red lines represents a damaged signal due to higher damping. c) fatigue crack detection with lamb wave [9]

In an ongoing development, active since 2013, Kurotec KTS have exploited the favorable properties of lamb waves to produce a non-destructive testing (NDT) method to monitor FRP

equipment. The sensors are coupled with an online device and uses SHM (Structural Health Monitoring) and CMS (Condition Monitoring System) to enable continuous monitoring of FRP-construction. The developed device (Figure L2) is attached to an FRP pipe, and from the sensor device a piezo crystal is connected and mounted onto the FRP-composite to generate and detect reflected lamb waves in the frequency range of 216 ± 50 kHz. The detection of relative changes in the material is performed in the connected sensor device by a custom-made analytical software. Each sensor can cover in total 6 m of FRP, 3 meter in both directions, assuming a construction with not to large diameter.



Figure L2: The sensor housing containing the radio sensor with a connected piezo crystal mounted on an FRP-construction. Figure L3: The gateway installed on the roof of the building communicating with the installed sensors and enable the data online.

A small micro controller radio sensor unit is built into the housing attached to the FRP equipment and a radio sensor sends the data from the housing to a gateway (Figure L3). The data is then transferred to the cloud and is accessible online. The distance between the sensor and the gateway can be long, so one gateway could thus cover a big area of the network and connect to several sensor units (Figure L4).



Figure L4: Smart network that enables online monitoring with a large number of installed sensors.

The micro sensor has a high efficiency and performance because of a long battery lifetime. From the software it is possible to get access to a lot of data, such as condition, visualization, data analytics, alert management and much more. In the dashboard, which is cloud-based, several properties can be monitored but for the case of failure prediction three signals are of extra interest. The signals are dynamic stress, static stress and degradation. The *static stress* is related to static loads, temperatures and other impacts, *dynamic stress* is the measure of e.g. oscillations from pumps, and *degradation* measure material aging, crack development and material damages. These values are compared with reference values (baseline values) and the deviation from the baseline is monitored, where material changes and stresses influence the propagation of the lamb wave. If the deviation is less than 50% from the baseline index the material degradation is considered acceptable. Any deviation greater than 50 % results in a warning from the monitoring software and alerts the operator that something is wrong. Two examples of this warning are shown below, where the first one report on a situation that occurred due to a falling tree in a storm. See Figure L5. When the tree fell on the pipe a change in stress level was detected and a warning was transmitted to the operator.



Figure L5. During a storm a tree fell on a pipe which was detected by the lamb wave sensor technology.

A second example comes from a leaking joint. During an extended time (ca 3 months) an increase in the dynamic stress region was noticed, which finally deviated outside the acceptable region, see Figure L6. The operator was alerted, and the pipe was de-mounted and investigated. It showed severe corrosion-cracks and blisters, which explains the change in signal (See Figure L7)



Figure L6. A steady increase in the dynamic region was seen for a time period of three months until the signal deviated more than 50 % from the baseline.



Figure L7. The online monitored pipe section showed severe material corrosion, which led to a changed signal for the lamb wave propagation.

3.5.2 Laboratory stack with sensors using the lamb wave technique

In a separate project running at RISE a new test bed (Figure L8) has been constructed that simulates the conditions in an incineration plant when a by-pass operation is conducted. A bypass operation means that hot flue gas, for some reason, cannot pass the ordinary cooling and cleaning step, but needs to be by-passed straight to the stack. The conditions in the stack, that is commonly built of FRP, will rapidly change (in a few minutes) from 70 °C and humid gas to 180 °C. This causes a great thermal stress on the FRP and can lead to the formation of delaminations (Figure L9-10) due to difference in thermal expansion of the different layers of the pipe wall. The aim for the test bed is to accelerate the formation of delaminations. If delaminations in a real stack are severe and influence the mechanical integrity of the structural layer of the FRP there is a risk that the whole stack needs to be replaced. Which can be very expensive. There is thus a need to improve the understanding why delaminations occur. So far it has not been possible to provoke such delaminations in a lab environment. The testbed is constructed so that industrially produced laminates with a cylindrical shape and a diameter of 40 cm can be tested for by-pass conditions in the lab. The air velocity in the test bed is up to 8 m/s and the heating and cooling from 60 °C to 185 °C takes 3:30 min respectively. At the lower temperature stage water in injected to raise the humidity of the circulating air.



Figure L8. Constructed setup for testing of FRP under conditions similar to a by-pass operation in a flue gas stack. The red arrows indicate the FRP test pieces and the white arrow the position where test coupons can be inserted.



Figure L9. Severe delaminations seen as large buckles in a flue gas stack. L10. Cross-section of a delaminating FRP from a flue gas stack.

3.5.3 Experimental

Since the test bed simulated real conditions in an incineration plant it works as a proof of concept for the investigated sensor techniques. In the investigation sensors were mounted on the two FRP test pieces to monitor material changes and stresses during cyclic cooling and heating in the test bed. The sensors were connected to a gateway, installed on the roof of the building (Figure L3), that transmitted the data to the cloud and made available on the online dashboard.

Two sensors were mounted in the test bed, one on the vertical pipe and one on the horizontal pipe. In Figure L11 the dashboard of the online monitoring is presented, displaying the status of the vertical pipe, battery level and scanning frequency. Measurement points have in this case been taken around 11 times per day. Unfortunately, the configuration of the used device did not allow for more frequent measurements. From the dashboard it can be seen that the status, for all three displayed categories, of the vertical pipe are green and OK (red box to the left in Figure L12). The monitoring has been running for roughly four months (130 days) with continuous heat cycles. Even though there have been no visible delaminations of the FRP-stacks some interesting data from monitoring the FRP-stacks can be seen.

In the dashboard the battery level, temperature and frequency of the specific sensor is visible at the top (red box). In the plot days are shown on the x-axis and on the y-axis the reference values of degradation (blue), dynamic stress (green) and static stress (light blue) are displayed.



Figure 28. The dashboard for the sensor on the vertical test piece. Status is green and OK (red box ti the left). Battery, temperature and frequency employed in the dashed box. The plot shows degradation, static and dynamic stress used to monitor structural health of the FRP-construction.

In Figure L12 the outside surface temperature (red), as measured by the sensor, of the vertical stack is added to the plot. From the plot it can be seen that the peak values for the other categories, degradation, dynamic stress and static stress, are all an effect of change in temperature. Due to the situation that measurement points are only taken roughly every second hour several of the heating cycles are not detected by the monitoring. The project of the test bed anticipated that the changes in temperature would lead to such stresses that it would consequently lead to delaminations. That it is possible to measure this stress as a function of the temperature is a success for the project. Between the 13th of June up to the 25th of July there is a slow but steady increase in the dynamic and static stress. It is unclear where this change is coming from, but it could be related to post-curing of the resin, material deterioration due to high temperatures or the formation of micro cracks.



Figure L12. The temperature (red) is added to the plot, and it can be seen that the temperature has a large influence on the response of the other parameters.

In Figure L13 the degradation, which measure material aging and crack development, can be seen in blue together with the reference value for the degradation (black). In the upper part of the plot the yellow field indicates the level defined as semi-OK, and the within the green field the status is OK. During the time of monitoring a slight deviation from the baseline can be seen up till the 25^{th} of July. Between the 21st and 29^{th} of May the test bed was run at 60 °C (except heating cycles). On the 29^{th} of May the test bed was turned off during the weekend and the temperature thus dropped, with a corresponding drop in degradation (indicated with red arrow in Figure L13). After the weekend (4^{th} of June) the temperature was increased to 70 °C and the degradation had a slight increased above the reference value, which was defined at 60 °C. The 4^{th} of June 5 holes where made from the outside of the FRP to insert thermocouples, unfortunately this cannot be separated from the effect of the temperature. On the 7th of June a 10 cm long and 4 mm deep slit and five 4 mm deep holes were made on the inside of the FRP-stack to give water access to the structural laminate. This can be the small step (black arrow) in the dynamic and static stress at the same time as the temperature is unchanged seen in Figure L14.

After the 25th of July the sensor, for some reason, re-calibrate itself and changed the level of degradation and reference value. After the 20th of August up to the 23rd of September the test bed was run constantly at 70 °C, which can be seen by the steady value of all measured parameters.



Figure L13. The degradation as monitored. Red arrows indicate positions when the test bed was turned off and thus had cooler temperature.



Figure 29. L14. The dashed lines indicate time positions when different damages were introduced to the FRP-stack.

In Figure L15 the generated data for temperature, degradation, dynamic and static stress for the period 16th of June to the end of September. The data of the horizontal pipe also indicate to be OK (dashboard not displayed) and the data seems, just like in the case of the vertical pipe, to be highly dependent on the temperature. For the case of the horizontal pipe the sensor calibrated itself before it was mounted on to the FRP-stack, this meant that the reference value once mounted was completely off. At the 29th of July the device calibrated itself again to reach a reference value closer to the vertical stack. For the horizontal stack no shift in the dynamic or static stress can be detected.



Figure L15. The monitoring of the temperature, degradation, static and dynamic stress over a time period of four months. No long-term changes in the material structure was seen during the time, even though it been cycled to temperatures above 180 °C more than 150 times.

Even though no delaminations have occurred in the test bed the lamb wave-based sensor seems to be a very versatile way to create smart FRP. By using the sensor

3.6 Ultrasonic testing for fibre reinforced plastics

3.6.1 Background

Ultrasonic pulse-echo (UPE) has been investigated as a technique to analyze FRP thickness, in order to predict the corrosion depth, delamination processes and/or if acid has penetrated the FRP. There are several limitations in performing a corrosion analysis with UPE since the signal is scattered and damped due to the high glass content in the material. For this reason, it is very important to choose the right operation parameters such as frequency, for the material investigated.

In this project the focus has been on using a small portable USB-stick-based US-device, see Figure 30, making it easy to carry and handle the equipment when performing field-analysis. However, the real break-through is the software developed to make it possible to create and process the raw data, thus making it possible to receive the crucial first echoes of the material. The main task is to optimize the method in order to receive as clear signals as possible. Pulse echo measurements are easy to perform in the field and the equipment is relatively inexpensive, portable and harmless to use. The major challenge lies in the signal processing in order to interpret the results from the measurements.

The next step is to create a library of different FRP signals. The third step will be to analyze FRPs with different corrosion conditions. The technique could then be implemented in the same device as the one used for the Lamb wave testing described above to give complimentary information.



Figure 30. The experimental setup for the ultrasound testing of samples using the small probe together with the USB software.

Ultrasound measurements are based on sending pulses of ultrasound into a material, and then measuring the time it takes for the sound to be reflected or transmitted in the material, see Figure 31. If the speed of sound is known in the material under investigation, the reflected or transmitted wave can be recalculated and interpreted as thickness. Different frequency ranges are used for different types of measurements; this is achieved by the choice of ultrasound detector. Higher frequencies are generally used for thin and homogeneous materials, lower frequencies for measurements of thick materials or materials that attenuate or scatter the ultrasound waves, for example GRP.

It is easier to measure the thickness of homogeneous materials such as metals and thermoplastics compared with GRP, which is a composite made up of several layers of a resin matrix and glass fibres with different characteristics. Various levels of glass fibre content and different additives might also change the velocity of the ultrasound wave and affect the measurement. If lower frequencies are used, the measurement becomes less sensitive to disturbance, but at the same time also less sensitive in the detection of small defects.

To increase the contact between the transducer and the test material a coupling medium (such as water, gel, etc.) is used.



Figure 31. Schematic picture of how ultrasonic measurements works.

One difficulty in traditional ultrasonic measurement of GRP material is its high damping. With the high damping and the fact that GRP is not a homogeneous material it can be difficult to get through thick material with traditional ultrasound transducers.

3.6.2 Experimental

In this project several different laminates of FRP were used for evaluation of the technique. First just the echo from a PMMA cube was recorded, see Figure 32. The PMMA cube is called a wedge and is used as a distance between the actual sample and the probe. The reason for using this distance is to be able to differentiate between the echo from the sample and the signals created by the surface/interface between probe and sample, the PMMA cube displaces the echo from the sample, as can be seen in Figure 29. In this way the signals are easier to evaluate since the signals from the FRP sample is being displaced from the signals from surface/interface disturbance.



Figure 32. The echo recorded from a PMMA cube.



Figure 33. The echoes from an FRP sample (to the left) and from an FRP sample with PMMA cube (to the right).

A sample of clear cast (with no fibres, just resin) was evaluated, see Figure 34. Here it is very easy to see several echoes and therefore the thickness of the sample can be calculated from these results. It should be noted that this sample has no fibres in it and therefore it is possible to see several echoes and thus calculate the thickness of the sample.



Figure 34. The echo recorded from a sample made of clear cast with no glass fibres.

The experiments show that it is possible to measure the thickness of FRP-samples with ultrasonic equipment if enough information about the laminate is known. The difficulty lies in knowing enough about the structure of the material investigated, glass fibre structure and resin, to be able to calculate the speed of sound in the specific laminate. If this is known it is possible to get the thickness. Defects in the materials, such as delaminations or blisters are hard to see with the ultrasonic device since this requires quite good accuracy and this can be demanding for the probes.

4 Discussion

There are several techniques that can be exploit in order to reach smart FRP. In this project the techniques that are more or less mature have been assessed. The most mature concept is the structural health monitoring using the lamb wave sensor developed by Kurotec. This system is under development but has proven to be an innovative way of creating a smart FRP that allows online monitoring. Considering that a sensor is needed every 6 meter it will requires many sensors to cover a hole factory plant. It may thus be more relevant to mount sensors on pipes and construction that are either difficult to manually inspect or critical pipes with high risk of failure. A major benefit with the lamb wave sensor is that no modifications of the todays FRP-laminates needs to be introduced. This means that the sensor can, by only fixation of the piezo crystal, with e.g. two-component glue, be added to any FRP structure, no matter the year of installation of the FRP-construction. Of course, the reference value will be set at the time of piezo crystal mounting, but any future material changes from that point will be monitored.

Even though no delaminations was provoked in the test bed during the time of monitoring, the technique gives an excellent way of following the experiment without being present. On and off mode, heat cycling, and structural changes can be followed just by logging into the homepage. Like in many of the other techniques one needs to increase the experience in the different responses of damages and failures. It would thus be of great interest to be able to follow a development of a delamination with the lamb wave sensor. This will hopefully be reached soon.

Electrical impedance spectroscopy is a non-destructive technique that, if correctly interpret can be a versatile method for monitoring corrosion. Carbon fibre strands can be added in between the corrosion barrier (CB) and the structural laminate (SL) during fabrication of the FRPconstruction. Since construction with CB and SL are often made in two curing steps, one for the CB and one for the SL, it should not be difficult to add the carbon fibre strand in between. Having the carbon strand in between the layers would make it possible to detect a signal once a solute has diffused through the CB into the SL, which is the dangerous zone for corrosive solutes. Besides that, FRP-structures are commonly manufactured with carbon fibres and the resistance to introduce them in the construction should not be too bad.

In order to take full advantage of the EIS-method more experiments need to be performed, both on the interpretation but also to identify frequencies that are not as sensitive to disturbance and can generate non-disturbed signals without the use of a Faraday box. In this case the most stable signals were found in the range $200 - 10\ 000$ Hz.

The InviSense humidity sensor is a concept that can be readily used. Since both the sensor and detector is already commercial products the task would rather be to utilize it and acquire more experience of its performance under different conditions. The RH-values of the sensor do also need to be further correlated to material changes in the FRP. The sensor can, just like in the case of the carbon fibre strand, be embedded between the corrosion barrier and the structural laminate. Thanks to the thin design, the sensor should be possible to incorporate with limited influence on the mechanical performance of the FRP-laminates. Collaborating companies have shown an interest to exploit the technique.

Since it seems as the cupper in the sensor is corroding when embedded in a clear cast there is a need to do some further test on the stability of the sensor, to verify that it works over long time periods.

In the case of the fibre bragg grating and resistance measurement more experiments needs to be carried out. For the resistance measurement a basic understanding of the corrosion rate when embedded in resin needs to be assured. The drawback with the resistance measurement is the introduction of metals into the FRP, this is something that there is a general resistance to since the metal acts as a defect in the laminate.

Ultrasonic measurements on FRP are very promising but the difficulty is that a lot of information about the material is needed in order to interpret the data. This is in many cases a problem since little is commonly known about the structure and laminate build-up of process equipment.

5 Conclusions

Several different sensor concepts have been investigated to create a smart FRP. The sensors based on lamb waves is considered the most promising method. It is also the method with a concept that is very mature. It has been, besides in this work, also evaluated in several other projects and industrial plants with promising results. The concept should be considered as a big step towards Industry 4.0.

The InviSense sensor is also a very promising method to detect water in FRP. The sensors work in a reversible mode and at high temperatures and the concept should be possible to implement with FRP-industry without too much work.

Electrical impedance is a very versatile and promising method to follow the diffusion. But more research is needed to correlate the dielectric response to physical and chemical changes in the laminate. If this is mastered the technique may very well turn out to be an important method to build smart FRP. The possibility to build in carbon fibre strands as sensor electrodes should be rather straight forward to implement.

Ultrasonic measurements of the thickness of laminates are possible if the exact composition of the laminate structure is known, but since this mostly is not the case, the technique needs to be developed further.

6 Future work

- Continue validation of lamb wave sensors
 - Study the influence of an ongoing delamination
- Improve the embedding of InviSense sensors and perform more exposures
 - Correlate the RH values to other methods
 - Perform exposures with other chemicals
 - Embed a sensor in an industrial produced FRP-construction to start a long-term experiment under industrial conditions
- More experiments of electrical impedance spectroscopy to correlate impedance response to physical and chemical changes
 - Construct a testing method for in-situ measurements
- For the ultrasonic testing more research must be done in order to develop this technique. The main difficulty is that the fibres in the laminates gives ultrasonic data that are very hard to interpret if the exact structure of the laminate is not known from the beginning.

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8 References

- [1] C. A. P. U. Johan Samuelsson, "Handbok för inspektion av glasfiberarmerad plast," Energiforsk AB, Stockholm, 2015.
- [2] C. a. m. r. i. K. Gunnar Bergman, "Alkali resistance of vinyl-ester-based FRP -Influence of temperature, concentration, type of surface veil, type of CSM and type of resin.," Corrosion & Metals research institute, KIMAB, Stockholm, 2006.
- [3] G. Bergman, "Long-term strength of GRP-laminates exposed to aqeous solutions at elevated temperatures," Swedish corrosion institute, Stockholm, 1992.
- [4] InviSense, "invisense.se," [Online]. Available: https://invisense.se/wpcontent/uploads/2019/02/White-paper-nr-1.1-English.pdf. [Accessed 30 September 2019].
- [5] H. S. Nalwa, Handbook of low and high dielectric constant materials and their applications, two-volume, Elsevier, 1989.
- [6] S. C. &. D. V. Moldoveanu, "Essentials in modern HPLC separations," Newnes, 2012.
- [7] D. C. Worlton, "Lamb waves at ultrasonic frequencies," Hanford atomic products operation, General Elctric, Richland, Washington, 1959.
- [8] Z. Y. L. &. L. Y. Su, "Guided Lmab waves for identification of damage in composite structures: A review," *Journal of sound and vibration*, pp. 295(3,5), 753-780, 2006.
- [9] M. Müggenburg, "NDT Device series.," in *FRP Unlimited 10th international Conference*, Munich, Germany, 2018.
- [10] P. A. S. David A, "Modeling the transport of low-molecular-weight penetrants within polymer matrix composites," *Appl. Mech. Rev*, 2006.

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