Advanced Non-Metallic Coatings and Composite Materials for O&G Industry

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Abstract. Materials used in the oil and gas industry are required to possess resilient properties to sustain operational challenges. These include high pressures and temperatures of the working fluids created by both reservoir depths and compressors combined with the inherent aggressive and corrosive components. Multiple solutions have been introduced to the industry to minimize corrosion probability in recent years. Utilizing external and internal tubular non-metallic coating as well as switching to the products entirely made of the non-metallic composites are the most commonly used and best performing solutions. This paper presents a comprehensive overview of the advances of the most promising research and development activities in the area of protective coatings as well as non-metallic composite material products with real case studies in downhole applications. Special emphases are put on the most promising technological breakthroughs such as 3D printing with non-metallic composites containing nano-sized short fibers and continuous nanofibers, which could be potentially used in the fabrication of future downhole composite products. The breakthrough solutions in composite technologies such as self-healing systems, including capsule-based, fiber-based and vascular healing network, which are perfectly aligned with the 4th Industrial Revolution are also described.

1. Introduction
Pipe corrosion is a common challenge for oil, gas, and petrochemical industries. Corroded pipelines must be addressing promptly to avoid leaks and any environmental concerns. Oil and gas wells are accounted to have high corrosive conditions since they operate in environments with high content of carbon dioxide (CO₂), hydrogen sulfide (H₂S) and chloride anions. Beyond these factors, there is also high acidity coming from wells’ acidizing as well as high salinity as the result of water injections. Stainless steel pipes historically utilized for oil and gas production and transportation are not best suited to withstand such severe operating conditions. Oil and gas production industry have been investing a lot of effort to monitor and handle corrosion issues [1-3]. Currently, there are a handful of technological solutions to resolve corrosion occurrence, including the use of corrosion inhibitors, Corrosion Resistant Alloys (CRAs), Inconel cladding, non-metallic coating deposition as well as switching to the non-metallic composite reinforced pipes, linings and casings.
Non-metallic (NM) materials primarily comprise of polymers and fiber-reinforced polymer composites. Polymers could be divided into three categories: thermoplastics, thermosets, and elastomers. Thermoplastics are easily processed by melting raw material and shaped by extrusion or injection molding. Thermoset is a highly crosslinked raw material that cannot be melted or reshaped after
consolidation. Thermosets generally constitute epoxy, vinyl ester and polyester resins. These materials have been well-known for their stability when exposed to high temperature, pressure and aggressive environment. Still, they were found to lose the performance at certain conditions. For instance, glass fiber–reinforced epoxy (GRE) products have been used in certain downhole applications with very limited in temperature rating (below 110°C). Also, these materials are subjected to issues, such as absorption of water, usually between 2 and 10% by weight, and susceptibility to chemical attack (usually hydrolysis) particularly at elevated temperatures. Hence, the thermoset materials are being recommended for shallower depths, with low temperature and low H2S concentration [4].

Elastomers are partially cross-linked polymers. They have the characteristics of rubber in terms of flexibility and elasticity. Elastomers are primarily used as o-rings, seals and gaskets in packers, completion equipment, liner hangers, plugs, logging tools, and alike [4-6]. Thermoplastics are major non-metallic materials that have high potential to be used in the oil and gas industry. The most promising thermoplastic resins used in downhole for high pressure and high temperature (HPHT) conditions are polyphenylene sulfide (PPS), polyvinylidene fluoride (PVDF) and polyetheretherketone (PEEK). High performance polymers such PEEK and PPS have equivalent mechanical properties and better chemical resistance compared to steel. Another important parameter of the thermoplastic materials is their superior properties of high chemical resistance at elevated operating temperatures [3,6]. These materials have excellent anti-corrosive properties, they can withstand relatively high operating pressures and temperatures, however they are not equal to stainless steel in mechanical strength and robustness. For this reason, reinforcement should be added to the final products.

Wide array of reinforcements could potentially be used namely glass fiber (GF), carbon fiber (CF), aramid fiber as well as fibers from natural sources such as basalt fiber. Carbon fiber is generally considered as the best reinforcement for high-pressure application due to its excellent mechanical properties. The major barrier to the massive deployment of advanced carbon fiber composites in the oil industry is the high cost of the material. Currently, the industry focuses on reducing the CFs cost and evaluating of the use of abundant renewable polymers, such as lignin or cellulose, and routinely recycled polymers, including polyolefins and polyesters, as feedstock for carbon fiber precursors [7]. Table 1 shows comparative mechanical properties of wide variety of common fibers used as reinforcements in composites [8, 9]

| Properties | Continuous Basalt | E-Glass | S-Glass | Carbon | Aramid |
|------------|-------------------|---------|---------|--------|-------|
| Density (g/cm³) | 2.63-2.8 | 2.54-2.57 | 2.54 | 1.78 | 1.45 |
| Tensile Strength (MPa) | 4100-4840 | 3100-3800 | 4020-4650 | 3500-6000 | 2900-3400 |
| Elastic Modulus (GPa) | 93.1-110 | 72.5-75.5 | 83-86 | 230-600 | 70-140 |
| Elongation | 3.1 | 4.7 | 5.3 | 1.5-2.0 | 2.8-3.6 |
| Max Temp (°C) | 650 | 380 | 300 | 500 | 250 |

In this paper, we report on the advances of the most promising research and development activities in the area of protective coatings as well as non-metallic composite material products with real case studies in downhole applications.

2. Non-Metallic Coating Solutions for Downhole Applications

2.1 Fusion-Bonded Epoxy (FBE), Liquid Epoxy and Polyurethane Internal Coatings for Steel Pipes

Thermoset-based coatings have been widely used in the oil and gas industry in the past decades. Primarily these coatings were heavily applied to the tubing and pipes externally, but they are also considered to be eligible for internal applications [10].

Epoxy-based polymers such as FBE are the widely used for gas/oil pipelines because they exhibit high chemical resistance, very low permeability to chloride ions, good mechanical flexibility, strong adhesion
to steel, and suitable processing characteristics, leading to improved corrosion protection and operational lifetime of the pipelines. The name 'fusion-bond epoxy' is derived from the way of resin cross-linking and their method of application which is different from that of a conventional liquid paint. FBE coatings are in the form of dry powder at normal atmospheric temperatures. The resin and hardener parts in the dry powder remain unreacted at normal storage conditions. At elevated temperatures, usually in the range of 180 to 250°C, the contents of the powder melt and transform to a liquid form. The liquid FBE film "wets and flows on to" the steel surface, on which it is applied, and soon becomes a solid coating by chemical cross-linking, assisted by heat. This process is known as “fusion bonding” [11]. The other type of anti-corrosion protective coating for steel pipes is spray-applied liquid coating. These liquid products are usually two component materials, consisting of an “A” and a “B” part. Typical spray applied coatings are made of epoxy, polyurethane, or a combination of both. Epoxy coatings are two-part, ambient temperature-cured, 100% solids (no volatile organic compounds), thermosetting materials with a base resin and curing agent. Polyurethanes are also two-part, 100% solids coatings but with a polyisocyanate curing agent and a polyol. Spray-applied epoxy and polyurethane coatings have excellent corrosion and chemical resistance properties. They are tough, resilient, and extremely abrasion resistant, making them a good candidate for internal coating [12,13]. However, epoxy coatings can easily absorb moisture, and the diffusion of absorbed water into the epoxy-steel interface owing to the presence of hydrophilic hydroxyl groups in the cured network (epoxy/amine reaction) weakens the interfacial adhesion strength between the epoxy and steel in a corrosive environment, causing system failure.

2.2 Polyamide (Nylon) Coating for Steel Pipes

Polyamide (PA) is an important thermoplastic NM material with amide linkage (–CO-NH–) in the polymer backbone. Polyamides are classified into various categories depending on the arrangement and chemical nature of monomers. Aromatic, cycloaliphatic, and aliphatic polyamides are important types of polyamides. Polyamide 6,6 (PA 66) and polyamide 6 (PA 6) are imperative types of polyamide. As these are aliphatic polyamides, they are also known as nylons. Nylon 6 and nylon 66 comprises about 50% of industrial polyamide consumption [15]. Polyamide 11 & 12 are the best fit NM materials to be used as steel pipe coating in the downhole conditions. The numbers represent the presence of 11 and 12 carbon atoms in the molecule respectively. Polyamide 11 is a polyamide derived from the vegetable oil and uses castor beans as the source. PA11 offers an ideal balance between the resistance of polyamides to grease and hydrocarbons and the resistance to acids, bases and salts of polyolefines [16]. Internal coatings based on polyamide chemistry are applied at very high temperatures and are then led through a controlled cool down process. Polyamide coatings generally require a liquid epoxy or phenolic primer in order to ensure good adhesion to steel.

2.3 Fluoropolymer Coatings for Steel Pipes

Fluoropolymers are the polymer materials containing fluorine atoms in their chemical structures. Fluoropolymers possess excellent properties such as outstanding chemical resistance, weather stability, low surface energy, low coefficient of friction, and low dielectric constant. Due to their special chemical and physical properties, the fluoropolymers are widely applied in the chemical, electrical, construction, architectural, automotive and oil production industries [16]. Polyvinylidene fluoride (PVDF) is a fluorocarbon chemical substance that is the homopolymer of 1,1-difluoroethene. It is characterized by high insolubility and extreme hardness, making it a good coating material for the prevention of corrosion in metal substrates. Polyvinylidene fluoride (PVDF) has been used for corrosive and chemical fluid containment since 1964. These materials were used as flexible tubing for large metal components. The PVDF copolymer systems allow for the creation of lightweight piping structures with excellent corrosion resistance and high flexibility or rigidity. PVDF may be used in a wide range of temperatures from -62°C to 149°C (-80 to 300°F). This property makes it suitable for the protection of metals exposed to drastic variations in temperature [17].
Ethylene tetrafluoroethylene (ETFE) is a thermoplastic copolymer derived from the polymerization of ethylene and tetrafluoroethylene monomers. These materials are extremely tough and abrasion-resistant having excellent chemical resistance and continuous operating temperatures up to 150 °C (302 °F). ETFE is also an excellent electrical insulator and has good nonstick and low-friction properties. In terms of heat resistance, ETFE can withstand prolonged heating (more than 1000 hours) at 200 °C without changing the properties. For a short time, in special cases, an operating temperature of 250 °C is allowed. In terms of chemical resistance to all known aggressive media, ETFE does not dissolve in organic solvents, it is resistant to concentrated acids, alkalis, and oxidizing agents [18].

3. Non-Metallic Composite Products for Downhole Applications

3.1 Glass Fiber Reinforced Pipes (GRP)

Glass fiber reinforced plastic (GRP) is a composite material that consists of a polymer matrix and glass fibers. The polymer matrix is usually an epoxy, vinyl ester, or polyester thermosetting resin. The resin brings the environmental and chemical resistance to the product, is the binder for the fibers in the structural laminate and defines the form of a GRP part. The glass fibers add strength to the composite. The most common type of glass fiber used for GRP is E-glass, which is alumino-borosilicate glass. E-CR-glass (Electrical/Chemical Resistance) is also commonly used in applications that require particularly high protection against acidic corrosion. The range of applications covered by GRP piping solution today is broad: from sewer systems and potable water lines to storage tanks, drainage pipes, industrial pipe systems, including linear tubing, production pipes and casing for oil and gas industries. Despite all the benefits that GRPs have, they face some challenges when used for oil production. The problems include poor mud cake removal, poor casing to cement bonding, problems with stability working at the HP/HT conditions [19,20].

3.2 Reinforced Thermoplastic Liners

Plastic liners have been used successfully to line and protect metallic onshore pipelines for many years and have become an indispensable requirement of the oil and gas industry, particularly with water injection and hydrocarbon services. In the case of internally corroded pipes, the use of thermoplastic liners for rehabilitation is an option to extend the lifetime of companies’ assets, reduce maintenance cost and increase testing and inspection intervals. For new construction, plastic liners in carbon steel pipes can compete technically and economically with pipelines of CRA materials and other corrosion inhibition systems. The cost of a liner is directly proportional to the operating temperature of the fluid. Pipelines containing fluid at temperatures up to 60 °C can effectively be protected from corrosion using high density (HDPE) or medium density (MDPE) polyethylene liners. Those having temperatures up to 100°C may be protected by Thermoplastic Polyurethane (TPU) or Polyvinylidene fluoride (PVDF) [21]. An innovative composite liner system that can withstand highly corrosive media at elevated temperatures has been developed by modifying its Kevlar core. This modification provides strong and flexible structure supporting an abrasion-resistant TPU exterior layer that contacts the damaged host pipe, and a corrosion-resistant polyvinylidene fluoride (PVDF) inner layer that is exposed to the corrosive service medium. PVDF material can withstand temperatures to 149°C and effectively prevent the pipes from corrosion [22].

3.3 Reinforced Thermoplastic Pipes (RTP)

Reinforced thermoplastic pipe (RTP), also known as flexible composite pipe (FCP) is a generic term referring to a reliable high strength synthetic fiber (such as glass, aramid or carbon) or high strength steel wire reinforced pipe system. Because of its expertise in producing pipes, Pipelife Netherlands was involved in the project to develop long length RTP in 1998 [23]. Another important benefit offered by an RTP is its spoolability, which enables it to be manufactured, packed on a road-transportable reel in a
long continuous form and transported to the site of the pipeline, with installation achieved by the reel-lay method. Consequently, the reel-lay method provides considerable cost and scheduling advantages, including low installation costs, and ease of storage and handling. During the past several years, RTPs have been used in both onshore and offshore applications, mainly as water injection pipes and oil flowlines.

The typical structure of an RTP consists of an inner thermoplastic liner, several structural reinforcing layers made from fiber-reinforced thermoplastic composites and an outer thermoplastic cover. RTP pipes have been used for the Exploration and Production (E&P) of crude oil and can operate in presence of wet and sour gases.

The Thermoplastic Composite Pipe uses glass and carbon fibres and PE, PA12 and PVDF polymers. The properties include: no corrosion, excellent chemical resistance, light weight, long length per spool, reducing installation cost, smooth inner bore combined with high external pressure rating, high internal pressure rating, and flexibility. TCP is qualified to 3000 meters water depth and is proven with track record in 2140 meters water depth [26].

M-pipe is a 100% composite pipe, comprising (approximately) 25% carbon fiber, 25% S2-glass and 50% PEEK, a construction that yields a pipe about one-tenth the weight of steel and traditional unbonded flexibles. Magma’s bonded m-pipe is manufactured by an automated tape laying (ATL) process, which fuses the alternating layers of glass and carbon in a PEEK matrix — a design which requires that flexibility be achieved through material selection and layup architecture.

4. Emerging Composite Technologies for Oil and Gas

Development of smart materials that potentially can autonomously eliminate corrosion and glue together the cracks formed at the micro- and nanoscale levels is one of the most promising for multiple industrial applications. These materials could be applied both for steel pipes’ coating and inside the products made of non-metallic composites. Smart materials can also be created in the form 3D printer filaments to create finished products from them used in oil and gas production. All these technological solutions can be attributed to the products of the fourth industrial revolution (4IR).

The term “fourth industrial revolution” was first coined in 2016 by Klaus Schwab, the founder of the World Economic Forum, in the book of the same name. In that book, the concept is defined as “creating a world in which virtual and physical production systems flexibly interact with each other on a global level.” The technologies of the Fourth Industrial Revolution, such as artificial intelligence, genome editing, augmented and virtual reality, robotics, intelligent materials and three-dimensional printing, are rapidly changing the way people create, produce and share values. Below we describe the advanced materials and products that we believe very well attribute to the 4IR concept and have a good chance to find application in oil and gas production.

4.1 Self-healing anticorrosive coatings and composite materials

With advances in research on non-metallic materials for coatings and composites, many researchers have reported smart coatings that can release the active components enclosed in them by some external stimulus. Intelligent coating can include various reactions, such as self-healing, self-cleaning, determination and elimination of corrosion. Among intelligent coating technologies, the concept of self-healing coatings has been introduced as a new approach to achieving the corrosion protection function. Self-healing functionality can be imparted by the presence of nano / micro containers of polymer and inorganic origin in the coating structure. The introduction of such smart coatings in many modern industries would protect the metal parts from the corrosive environment, improve the performance and ensure safety [27].

Significant number of publications were devoted to inducing self-healing properties to the epoxy-based coatings with the help of microcapsules. This material has broad applicability to the oil and gas production as it was presented in the previous part of publication, so making it self-healing could benefit stability of crude resources’ production. The most efficient self-healing actives for oil and gas would be the ones that can react upon exposure to moisture due to the downhole environment. One example of
this material could be silyl esters do not require catalysts or cross-linking for polymerization (or solidification) because, in their case, barrier formation is based on the hydrolysis process. Silyl esters show good reactivity with both water and metal; this allows them to form an adhered-to-metal barrier that can prevent corrosive materials from encountering the underlying metal [28]. García et al. [29] fabricated an epoxy matrix composite, wherein 100-μm urea-formaldehyde microcapsules filled with silyl ether were embedded in the epoxy. The results of the impedance and current density measurements obtained in the case of the microcapsule-embedding composite confirmed that it provided for anticorrosion performance as compared to that of the bare samples.

Isocyanates represent another excellent candidate for self-healing for epoxy-based products in downhole conditions. As silyl ethers they also react with water and form protective layer on the metal’s surface. However, the water-friendly properties of the isocyanate group can cause issues during encapsulation in a shell polymer, given its high reactivity. In order to prevent these possible issues, Wu et al. [30] fabricated polyurea/silica hybrid microcapsules for encasing isocyanate-based healing agents by combining interfacial polymerization and an in-situ sol gel process. The hybrid structure of the microcapsules resulted in improvements in the thermal and chemical properties of the microcapsules. As a result, during a corrosion test, wherein the samples were immersed in a 10 wt% NaCl solution for 48 h, the hybrid-microcapsule embedding epoxy resulted in a completely rust-free substrate. Following the same research rout, Sun et al. [31] fabricated 88-μm double-layered polyurea microcapsules. They could successfully encase an isocyanate-based healing agent within microcapsules. The resulting microcapsules demonstrated impressive stability in non-polar solvent as well as when exposed to the elevated temperatures. Both conditions very well represent the environment in the downhole.

Another option to induce self-healing to non-metallic coatings and composites is by embedding micro- or nanosized fibers filled with adhesive inside the non-metallic matrix material. This technological approach is called vascular-type self-healing. The fibers could be made by several methods, one of the most frequently used for nanofibers (NF) synthesis is electrospinning. Electrospinning is the method that allows to fabricate networks of core-shell nano- and microfibers that are filled with enough healing agent. As the example of this technology, core-shell nanofiber coatings were prepared on steel substrate with dimethyl siloxane (DMS) as self-healing agent and dimethyl-methyl hydrogen-siloxane (as curing agent) separately in the cores via the dual emulsion electrospinning method. DMS and dimethyl-methyl hydrogen-siloxane were both encapsulated in polyacrylonitrile (PAN) shells. These dual nanofibers were deposited onto a steel substrate. Finally, the nanofiber mats were intercalated with poly (dimethyl siloxane) (PDMS) matrix. The corrosion experiments that were conducted on the manually damaged nanofiber coating proved the self-healing efficiency and corrosion resistance of these coatings [32].

Self-healing property could be induced not just to the epoxide coatings, but also to more advanced engineered plastics. Liu et al. demonstrated the possibility to create self-healing PVDF coating, using porous Na2CO3 particles grafted by nano-SiO2 to store the healing agent of fluorinated silane (POTS). Besides the self-healing capability this coating demonstrated superhydrophobic properties, scratch resistance and stability when exposed to acid and alkaline solution [33].

### 4.2 3D printing with composites

For oil and gas production, 3D printing must be performed using high-performance engineering polymers. One of the most well-known materials that can withstand high temperatures and aggressive environment is an amorphous thermoplastic polyetherimide (PEI) material. It can operate at 160-170°C while maintaining strength and rigidity.

Polyetheretherketone (PEEK) is another outstanding material that can be applied for 3D printing. Latest research in the area of using PEEK for 3D printing is focused towards laser sintering of the PEEK powder. Leading laser sintering system supplier EOS offers its own certified PEEK material, making it the only current provider of a readily approved advanced thermoplastic powder for its own polymer powder bed fusion technology. More recently, the industry was able to produce high-performance polymer and reinforced with carbon fibers. Currently, 3D printers are able to mass produce high-temperature poly-ether-ketone-ketone (PEKK) polymer functional parts [46].
3D printing composites are extremely beneficial when making lightweight yet strong parts. The fibers add strength to a part without adding weight. There are two types of reinforcements, short fiber or continuous fiber. In the first case, chopped fibers, which consist of segments less than a millimeter in length, are mixed into traditional thermoplastics to increase the stiffness and to a lesser extent the strength of components. Chopped fibers can be mixed with various thermoplastics like PEI, PA, PEEK, PEKK. Short fiber composites can be extruded in the regular 3D printing with filament process since the filament already contains the fiber.

On the other hand, continuous fiber 3D printing is a trickier process that requires two nozzles to print at the same time. The process for making continuous fiber composites parts is not as easy as short fiber composites parts because the fibers need to be integrated into the thermoplastic continuously as the thermoplastic is being extruded. There are two known types of 3D printing using continuous fiber: the first type is printing with UV-cured thermoset materials while the second is extruding the thermoplastic material from one nozzle and the continuous fiber from the other. The UV-cured thermoset method is an automated process that leverages the true anisotropic properties of a continuous fiber by printing in the direction of the load conditions. This eliminates the need for molds, ovens, and autoclaves, resulting in high material yield.

There is a big variety of 3D printer producers working with the second method, using thermoplastics coming from one nozzle and continuous fiber from the other. One way to implement this method is by using a technology called Directed Energy Deposition, in which a laser is used to heat the filament and carbon fiber at the same time as a roller compresses the two together. Another process to achieve the thermoplastic material extrusion method is by using a process called Composite-Based Additive Manufacturing (CBAM). In this process, sheets of fibre reinforcement material, such as carbon fibre, are passed beneath an inkjet printhead, which deposits a liquid solution onto the sheet, in that layer’s shape. Then, the sheets of carbon fiber are weaved into a print by using a lamination process. Because of using inkjet printing, the CBAM method is much faster than extrusion processes, and there’s also the possibility to print large parts. Some of these 3D printers can work with high performance plastics PEI or PEEK. In the oil industry, 3D printing using non-metallic materials, especially those filled with short or continuous carbon fibers, can be implemented for a wide variety of products and spare parts [34].

5. Conclusion and Path Forward Strategy

Promoting the deployment of the nonmetallic composites in oil and gas and minimizing the life cycle cost (OPEX) are key objectives that the industry is diligently realizing by accelerating the deployment of proven and emerging technologies in water injection/disposal and shallower hydrocarbon wells. Deployment of the nonmetallic materials and composites is very strategic to the oil industry as it allows to significantly reduce or even negate corrosion impact, increase oil demand and reduce the energy consumption. Moreover, the flexibility of composites helps improving the integrity of the intervention and drilling operations in the ultra-deep directional application and potentially minimizing downhole friction and lockup. Ongoing collaborations with the R&D entities promotes the increase of the reliability and expand the operation envelope for cost-effective materials to withstand harsh environment in extended reach oil/gas wells. The path forward strategy is complete support of the R&D activities in the area of nonmetallics and composites for more demanding HPHT applications including drilling, tubulars and completions.

The following areas require further R&D efforts to overcome challenges present in the downhole:

- Support the nonmetallic materials and composites' standards development for downhole
- Introduce the self-healing functionality to the non-metallic coatings and composites for downhole applications
- Introduce the additive manufacturing and 3D printing technology for downhole testing
- Reduce the carbon fiber manufacturing cost and account for its recyclability
- Develop and implement cost-effective reinforcements from natural resources, such as basalt rock and date palm fibers
• Support the development of the intelligent composite products by embedding a sensing feature such as fiber optic, piezoelectric, etc. for in-situ monitoring of the temperature gradient, possible strains and cracks.

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