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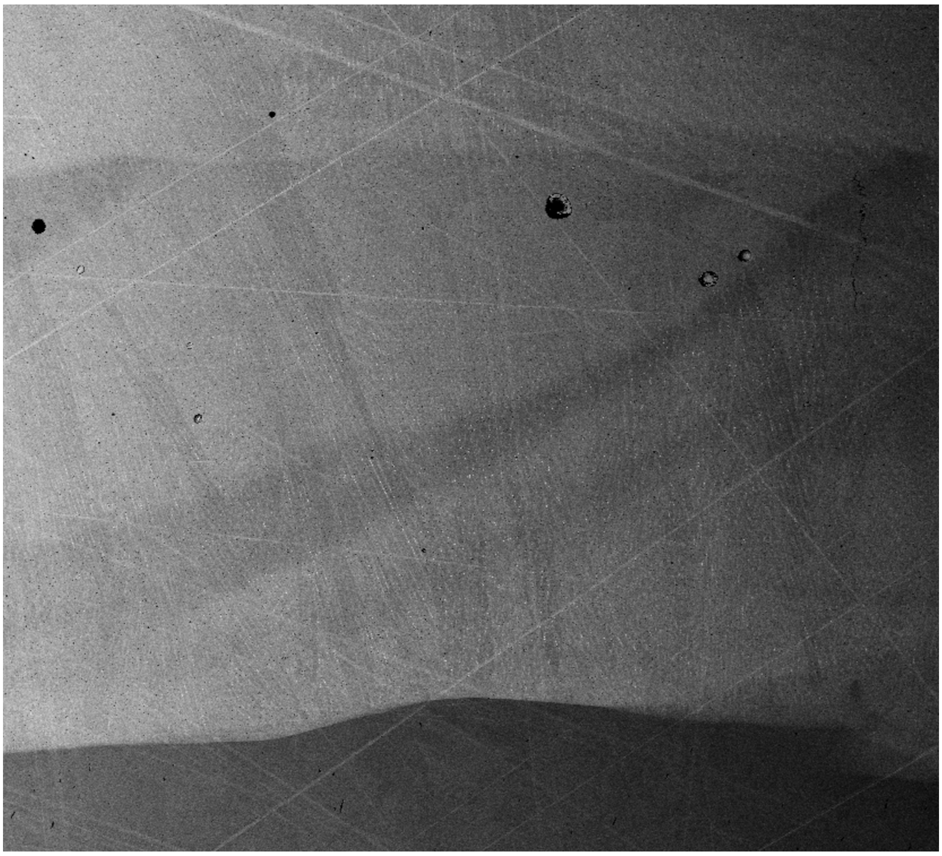

Industrial application of metal coating for improved corrosion resistance

A study of coating possibilities of a stainless steel flange for increasing corrosion resistance

Master's thesis in Materials Engineering

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Gothenburg, Sweden 2022
www.chalmers.se

Master's thesis 2022

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Cover: SEM micrograph of the interface of the Ni-alloy clad stainless steel flange.

Typeset in LaTeX
Printed by Chalmers Reproservice
Gothenburg, Sweden 2022

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Abstract

Gradual degradation of metal surfaces also known as corrosion is a well-known phenomenon found in most industries. This destructing mechanism can lead to reduced utility and efficiency, or in the worst case, total failure of plants, equipment, and components which is extremely costly and also serve consequence to human life and the environment. An industry that is greatly affected by this problem is the oil and gas industry where crude oil and other aggressive chemicals are common. To ensure that all processes safely take place, these are continuously checked by different types of level switches and systems that are usually mounted with flanges on tanks and pipelines. Which makes them exposed to corrosion.

This report investigates the possibilities of increased corrosion resistance of stainless steel flanges with help of cladding. A prototype was developed by initial material- and process selection for the cladding which then was verified by different laboratory tests to examine the final properties of the prototype.

The results gained from the laboratory tests of the prototype show that the cladding continued to have good corrosion resistance even after welding, as well as good adhesion with the substrate with few defects in the interface which means a good balance in the amount of dilution between the different materials. However, the cladding showed porosity which was expected according to the manufacturing method. These results are a good indication that cladding can be a good method for increasing the corrosion resistance of flanges.

Keywords: corrosion, cladding, flange, oil and gas.

Acknowledgements

First of all, I would like to express my gratitude to Emerson Automation Solutions for giving me this opportunity to work on such an interesting and important topic.

I also extend my gratitude to my supervisors Bengt Hägglund from Emerson Automation Solutions and Lars Nyborg from Chalmers University of Technology. The time at Emerson under Bengt Hägglunds guidance has been very educative and I feel very grateful for his guidance and the effort. Lars Nyborg's feedback and guidance throughout the hole process has also been vital for this thesis.

Lastly, I would like to give a special thanks you to my family and friends for their continuous support during this master thesis work.

Antonio Butkovic, Gothenburg, May 2022

List of Acronyms

Below is the list of acronyms that have been used throughout this thesis listed in alphabetical order:

API	American Petroleum Institute
ASME	American society of mechanical engineers
CO_2	Carbon dioxide
EDS	Energy-dispersive X-ray spectroscopy
GTAW	Gas tungsten arc welding
HAZ	Heat affected zone
H_2S	Hydrogen sulfide
ISO	International Organization for Standardization
NACE	National Association of Corrosion Engineers
SEM	Scanning Electron Microscopy
TS	Thermal spraying

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1
Introduction

The following chapter will explain the relevant background to the thesis. A brief introduction to the case company is given, followed by the purpose of the master thesis along with the research question and limitation

1.1 Background

Gradual degradation of metal surfaces is a well-known phenomenon that is a natural costly process of destruction. The common mechanism associated with this type of phenomenon is corrosion or wear. These two types of destructing mechanisms can lead to reduced utility and efficiency, or in the worst case, total failure of plants, equipment, and components which is extremely costly and also serve consequence to human life and the environment. The global cost due to corrosion reaches as much as US$2.5 trillion in 2013[1]. Some of the biggest costs due to corrosion is the oil and gas industry, automotive industry, and aircraft industry. Where the offshore oil and gas industry alone stands for US$60 billion[2]. Since this is a very costly problem for many types of industries and is a major contributing factor to the constant development of new technologies to prevent this. This includes the development and use of a coating to improve their performance in specific contexts. This results in a significant increase in the service life of materials and equipment and contributes to less maintenance.

Corrosion will occur in all types of environments to some degree, and the corrosion rate is directly affected by the environment. A tougher environment like acids, alkalies, and soils will provide a higher corrosion rate compared to fresh water or a marine environment. One of the toughest places where the materials are exposed to some of the most aggressive industrial environments is the offshore oil and gas industry, especially the pipeline and flange connections which are exposed to aggressive chemicals. Flanges for the offshore industry are typically made of duplex or stainless steel which generally has good corrosion resistance in most environments but not all. When it comes to tougher environments, such as aggressive chemicals the flange connections are normally made of superalloys for example Nickel-alloys which are much more resistant and expensive[3].

A possible solution to enhance the corrosion resistance and increase the longevity of metallic structures is to isolate the substrate from contact with the surrounding environment, also known as a coating. This means that the entire flange does not have to consist of an expensive super alloy just only a thin layer is enough to protect the surface of the flange. Since there are several available coating systems and materials it is important to carefully pick the most suitable for the application. To make the right choice of the coating process and material several constraints are set for the application, which will be discussed later.

1.2 Case Company

The case company is a part of an innovative multinational corporation that manufactures products and provides engineering services for industrial, commercial, and customer markets. The case company is structured into two business units; automation solutions, and commercial and residual. The automation solution area offers products such as measuring and instrumentation, integrated solutions, software and services which enables maximizing production, protecting personnel and environment, etc. Common applications for these types of solutions is

- Gas and oil industry
- Pulp and paper
- Marine
- Mining minerals and metals

The other segment; commercial and residential solutions provide products such as compressors, condensers, heat exchanges and services to create comfortable and controllable workplace environments. Common applications for these types of products are heating and air conditioning, refrigeration, food waste disposals, etc[4].

1.3 Purpose and research question

As mentioned in the background, the corrosion phenomena are presented as a major problem that is costly for a lot of industries, especially for the offshore oil and gas industry where the pipelines and flange connections are exposed to aggressive environments. This leads to low service life of components and a high maintenance rate to prevent a catastrophic failure that serves consequences to human life and the environment. Today, there exist engineering materials that are extremely resistant to corrosion in aggressive environments, but they are also very expensive. Too expensive to manufacture complete components from it, therefore it is interesting to investigate alternative solutions to enhance the improved corrosion resistance of flange connections. The purpose of this study is therefore to:

Investigate the possibilities of using coating as corrosion protection for stainless steel flanges in a Offshore environment through developing a prototype and validate it by laboratory tests.

In order to meet and fulfill the purpose of this study, an understanding of which requirements set from the given application the coating has to meet is needed. Also, an understanding of all the common coating systems and suitable materials which has possibilities to fulfill the requirements of the application are required. The purpose is therefore decomposed into the following research questions:

RQ1: What requirements are set by the industry regarding corrosion protection with coating?

RQ2: Which materials and coating methods are best suited for corrosion protection?

RQ3: How does the selected coating perform in the test requirements set by the industry?

1.4 Limitations

The following section will outline the limitations of this master thesis and clarify which areas will not be examined. This problem can be divided into 3 parts, material, production, and construction problem. This study will focus on the material and production part which includes finding a suitable cladding that meets the requirements for corrosion, coating compatibility, and industry standards. The work will not focus on the construction part which includes redesigning the flange for optimized cladding performance, the cladding will be performed and studied on an existing flange from the case company's product range. The choice of material and method for the corrosion protection will be based on the current construction of the flange. The study will not focus on details in the manufacturing process of the coating, such as optimal cladding parameters.

1.5 Thesis outline

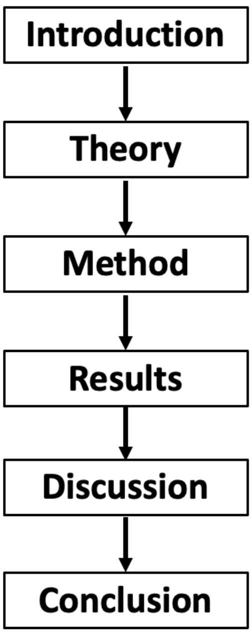

Figure 1.1: Structure of report.

Theory
This chapter gives the theoretical background of this study. The chapter begins with an overview of different coating technology and how is it beneficial for improving the performance of flanges. The background of the oil and gas industry is given together with all the requirements that must be fulfilled.

Method
This chapter describes the methodology of this study where all the steps are described in detail. The chapter begins with a description of how the literature was chosen and conducted. After that, the selection of coating process and material are described in detail. Lastly a detailed description of how the laboratory tests were performed on the prototype.

Results
The results have the purpose to describe which coating method and material is most suitable to use. These results are based on theory and the constraints set by the standards of the industry. This section also presents the results from the laboratory tests of the developed prototype.

Discussion
This chapter will discuss the results from the theoretical study and the analysis of the developed prototype. Also, the research questions will be answered, followed by a discussion of the limitations and future research considered in this study.

Conclusion
This chapter will summarize and conclude the findings that have been made in this study.

2

Theory

This chapter includes the theoretical background upon which this study is based upon.

2.1 The Oil and Gas industry

Crude oil is a natural resource that occurs naturally in parts of the bedrock. This thin dark liquid has been used by humans for thousands of years in many different applications. In modern times, it is mainly used to produce various materials such as polymers but also in the production of various fuels. Crude oil itself is not corrosive, but often crude oil consists of contaminants that can cause corrosion. The main corrosive constituents which arises during production within the oil and gas industry is CO_2 and H_2S. This constituents can cause localized types of corrosion attacks such as pitting and crevice corrosion and the corrosion rate increases initially with an increase in its partial pressure and temperature.
The oil and gas industry is a large industry that is usually divided into three different sectors upstream, midstream, and downstream. Where the upstream sector consists of localization and extraction of crude oil. This can be done offshore, also called offshore, or on land which is done on an oil rig. The midstream sector handles and transports crude oil to various extensions. Finally, the downstream sector where crude oil is processed into various products such as fuels, plastics, etc. The difference between the upstream and downstream sectors is service conditions such as temperature, pressure, and chemical additives. Where the downstream sector is more controlled and where these parameters can be controlled compared to the upstream sector which is uncontrolled.[5], [6].

2.1.1 Coating requirements and desired properties of a flange

To prevent unpredictable disasters that can affect the population and the environment-specific standards are set in most industries. These standards are usually created by a combination of experience and expertise of all intrested parties in the industry which is then controlled by national governments, and organizations such as the UN, etc.

Standards are extra important in the oil and gas industry as this industry largely consists of toxic substances for both the population and the environment. As these toxic substances are commonly transported over long distances, usually under the ground and under the seabed, it is important to avoid failure such as leaks, cracks, etc. which would have enormous consequences. Some common standards in the oil and gas industry are:

- Norsok
- API
- ISO
- NACE
- ASME

To be able to make an optimal material and process selection, reduce the risk, decrease inefficiency and meet higher performance levels, these standards must be taken into account[3], [7].

The desired properties of the flange, like toughness, weldability, high strength, stiffness, corrosion resistance, erosion, ability to withstand high pressure from the tank, and others properties are studied. As this study will focus on coating a standardized flange from the case company's product range only, the corrosion resistance and weldability will be in focus, the other properties will not be taken into account in this study. The coated flange must possess some of the desired properties like:

- have excellent corrosion resistance against aggressive substances such as CO_2, H_2S in a marine and industrial environment.
- be weldable with the probe which connects the tank with the measuring system.
- the coating process have to be conventional

These stated desired properties will be set as constraints for the further material and process selection of the coating.

2.1.2 Flange material and design

The flange is a common and important part of the oil and gas industry and can be found throughout the whole production chain. The main purpose and the most common application of a flange is the connection and fastening of pipelines, vessels, containers, etc. An also common application of a flange is to fastening different types of measuring devices to be able to control different parameters in the pipelines and tanks to make sure that all runs safely.

In the oil and gas industry, the flanges are mainly manufactured in stainless steel, cast iron, aluminum, brass, super alloys, etc. Stainless steel is a popular and commonly used flange material due to its good corrosion resistance and cost-efficiency. However, the stainless steel can not withstand all types of aggressive substances and when the process consists of aggressive elements such as chlorides, sand, organic acids, carbon dioxide, and hydrogen sulfide in combination with high pressure and temperature the flange has to be manufactured in super alloys to be able to withstand these areas and decrease the corrosion rate, and this is rather expensive. Some solutions consist of a superalloy plate that is mounted or welded between the flange faces to protect the flange from the internal aggressive substances, and this is more cost-effective compared to the solid super alloy flange. Depending on the application, these flanges are designed in different ways to suit the purpose. Some examples of common flange types are welding neck flange, socket weld flange, lap joint flange, etc. Because flanges can be designed in so many different ways, it is not always easy to manufacture these flanges in superalloys. With cladding, a lot of possibilities will be opened up from a construction point of view, and a wider range of flanges will be able to use in tough environments[8].

2.2 Types of corrosion

Corrosion is a costly and dangerous phenomenon in the oil and gas industry that causes material degradation of important components in the oil and gas production and transportation facilities. Crude oil and gas can contain various amounts of highly corrosive media, such as H_2S, CO_2, and free water. Together with the marine environment, it creates harsh conditions for most materials. The combination of the aggressive substances, harsh environment, and high temperature and pressure cause a material degradation which results in loss of mechanical properties like strength, ductility, impact strength, and so on. This leads to loss of materials, reduction in thickness, and at times ultimate failure[5].

2.2.1 Hydrogen sulfide corrosion

Hydrogen sulfide (H_2S) is a common contaminant in crude oil. This toxic gas is usually released by the decomposition of sulfur compounds in crude oil. This occurs when sulfur comes in contact with water at high temperatures. As hydrogen sulfide is easily soluble in water, it forms an acidic solution, which is corrosive to many different types of metals. This acidic solution causes an acidic type of corrosion which causes sulfide stress cracking and affects most materials.[9], [10].

2.2.1.1 Hydrogen sulfide corrosion mechanism

Corrosion due to H_2S is associated with stress corrosion cracking (SCC). Stress corrosion is caused by the combination of a corrosive environment and external or internal stress creating a crack in the surface of the metal. In an environment consisting of H_2S, the absorption of hydrogen atoms by the metal usually enhanced. When this happens, the hydrogen atom diffuses easily at ambient temperature. This usually occurs in places where high-stress concentrations occur, for example, grain boundaries and inclusions. These lead to these places becoming brittle and initiation of brittle crack formation, when the material is subjected to tensile stresses[10].

2.2.2 Carbon dioxide corrosion

CO_2 corrosion or "sweet corrosion" as it is also called it's a common type of corrosion that causes metal degradation in the oil and gas industry. CO_2 is a stable, inert, and non-corrosive gas in temperatures occurring in oil and gas. However, when CO_2 is dissolved in water it forms H_2CO_3 and becomes corrosive due to the electrochemical reaction between steel and the aqueous phase[11].

2.2.2.1 Carbon dioxide Corrosion mechanism

When CO_2 is dissolved in water it forms H_2CO_3, this usually causes general and pitting corrosion. Pitting corrosion is a type of corrosion where cavities and "holes" are produced on the material surface. This usually occurs at low flow rates and where the destructive process usually starts through local breakdown of the passive surface through a specific interaction between the film and the corrosive ions in the environment. The layer often fails at spots of the metal where there is a high degree of cation vacancy diffusivity. The effect of this type of corrosion increases with temperature and pressure[11], [12].

2.3 Coating

In order to achieve requirements that are set in the development of products such as appearance, mechanical properties, availability, cost, recyclability, toxicity, etc. there are today different methods available such as heat treatment, alloying, and coatings. Among these solutions, coatings are the widest and are available in many different forms. The coating is thus a thin layer of a specific material that encloses another material. Where the coating is intended to protect the underlying material and thereby increase the life and durability of the material. There are two major groups of coating methods, cladding, and plating. The difference between these two techniques is that cladding gives as a layer that is metallurgically bonded to the substrate, and this generally forms a quite thick layer. Plating on the other hand is a material layer that is electrochemically deposited to the substrate and gives a much thinner coating compared to cladding. In these two groups, there are several techniques available for coating a surface, and the most common are[13]:

- Physical Vapor Deposition (PVD)
- Chemical Vapor Deposition (CVD)
- Electrodeposition Coating
- Thermal spraying
- Plasma spraying
- Laser cladding
- Cold spraying

According to the desired properties stated in section 2.1.1, this study will focus on Thermal spraying, laser cladding, Cold Metal Spray, Tig welding, and Mig welding.

2.4 Laser cladding

Laser cladding is a common technology used in industrial applications such as repairing, coating, developing alloys, and more. The technology is based on the that a feedstock material is melted down with help of a laser beam as a heat source. The feedstock material can consist of either powder or wire that is injected and builds up thin layers on the base material. The energy source melts down both the feedstock material and the base material, which creates a melt pool where the different materials bind to each other with a metallurgical bonding. Figure 2.1 shows a schematic of laser powder cladding. Laser cladding is a low-temperature process compared to other cladding processes with a temperature around 500°C, this result in a relatively low dilution between the base- and feedstock material. This is positive, as the coating layer is not affected by the base material and the coating material retains its properties. But this can also be negative when it comes to the attachment between the base material and feedstock material[14].

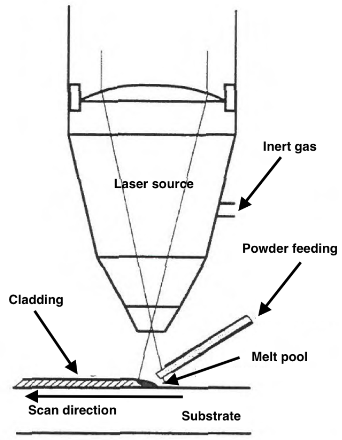

Figure 2.1: A schematic of laser powder cladding, modified from [15].

The quality of the developed clad can be assessed by different factors, such as microstructure, crack formation, adhesion between the clad layer and base material, chemical composition, and hardness variations along the cross-section[16], [17].

2.4.1 Powder vs wire as a feedstock

The dilemma between powder or wire as feedstock material has been discussed for a long time when it comes to quality and efficiency in laser cladding.

Several factors can affect the quality of a powder, such as particle size, distribution, particle shape, and internal porosity. The particle size and distribution are crucial, a smaller particle size of the powder requires a lower heat input for melting. This results in a smaller heat affected zone (HAZ) and a lower dilution between the base material and cladding layer.

The quality of a wire feedstock on the other hand depends on the composition. The other properties, such as diameter, stiffness, and surface quality are easier to control. In this regard wire as a feedstock is much more consistent compared to powder which can differ in quality. However, the powder is still the most common feedstock option when it comes to laser cladding. This is because wire has a small surface area with a reflective surface, which makes it hard to melt which leads to highly diluted cladding[18], [19].

2.5 CMT Process

CMT stands for Cold Metal Spray and is a modified MIG/MAG welding process that utilizes short-circuit transfer. It is a welding process with low heat input compared to the MIG/MAG. The difference between CMT and MIG/MAG lies in the droplet detachment combined with the electrode movement. The digital process detects a short circuit and the wire is pulled back to assist in the drip transfer, which is illustrated in figure 2.2. In this way, the arc feeds heat very briefly during arc formation, and then the heat supply is quickly reduced when the wire pulls back. This results in a controlled method of material deposition and low thermal input[20].

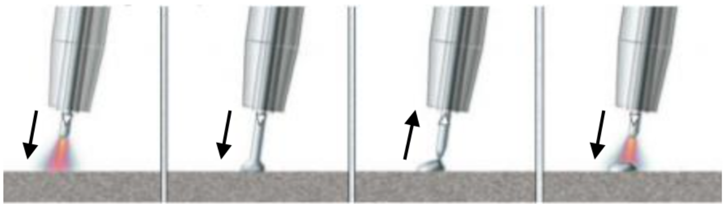

Figure 2.2: A schematic of CMT process, modified from [20].

The resulting low heat input of the CMT technology makes this method obtain a low dilution rate compared to the conventional MIG/MAG and TIG welding.

2.6 Gas Tungsten Arc Welding (GTAW)

GTAW also known as TIG welding is a process that has been used for decades for joining dissimilar metals, such as aluminum and magnesium. The process consists of a welding gun that provides an arc between a pointed tungsten electrode and the work piece, as can be seen in figure 2.3. The area is shielded by an inert atmosphere, helium or argon. The filler material is added separately by an electrode. It is a high-energy process with a temperature of around 3000°C, resulting in the melting of both the filler and substrate material. This gives a large heat effect on the substrate in the form of a large heat affected zone (HAZ), as well as a high degree of dilution of substrate and filler material. The high heat input provided from the process leads to good bonding between the base material and filler material in form of metallurgical bonding[21].

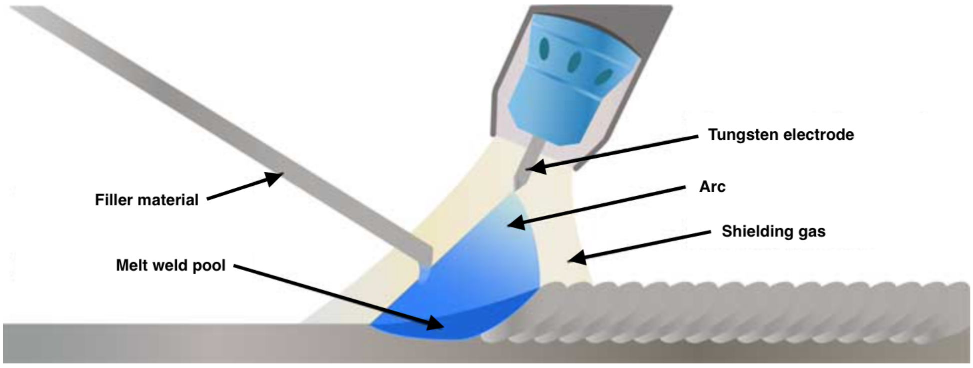

Figure 2.3: A schematic of GTAW process, modified from[21].

2.7 Thermal spraying

Thermal spraying (TS) is a general name for a family of coating processes used to apply molten or semi-molten droplets of a specific material to produce a protective coating. The method is widely used due to its possibility of a wide range of deposit materials, such as metallic, composite, cermet, and non-metallic coatings.

In the TS process, the spray material can be fed in form of powder, wire, or a rod into a spraying gun where it is heated up by a heat source. The melted feedstock material is then accelerated by a stream of gas which often consists of oxygen and fuel. When the mixture ignites the resulting detonation heats and accelerates the feed material through the spray gun with a supersonic velocity. The high kinetic energy molten particle's hit the substrate surface, the big impact deforms the base material and creates a mechanical bonding between the substrate and the coating particles. This results in a thin and strong coating[22]. The basic principle of the TS process is shown in figure 2.4.

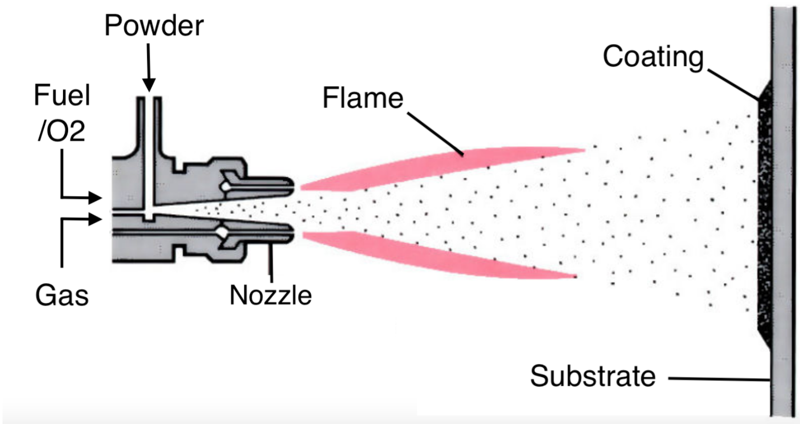

Figure 2.4: Schematic illustration of the TS technique, modified from[23].

2.8 Mechanical- and metallurgical bonding

For the material to be able to maintain its mechanical properties after the coating process, good adhesion between the coating and the substrate is required. Two common bonding mechanics are metallurgical and mechanical bonding.

2.8.1 Metallurgical bonding

Metallurgical bonding is a type of chemical bonding which is obtained between dissimilar metals by transferring or sharing electrons. This type of bonding is a result of the melting of both the base material and cladding material, also called dilution which causes a metallurgical change in the coating and substrate interface. This gives a very strong bonding between the substrate and coating. This can be seen in figure 2.5. This also causes a thermal gradient in the base material, which can affect the microstructure and properties[24].

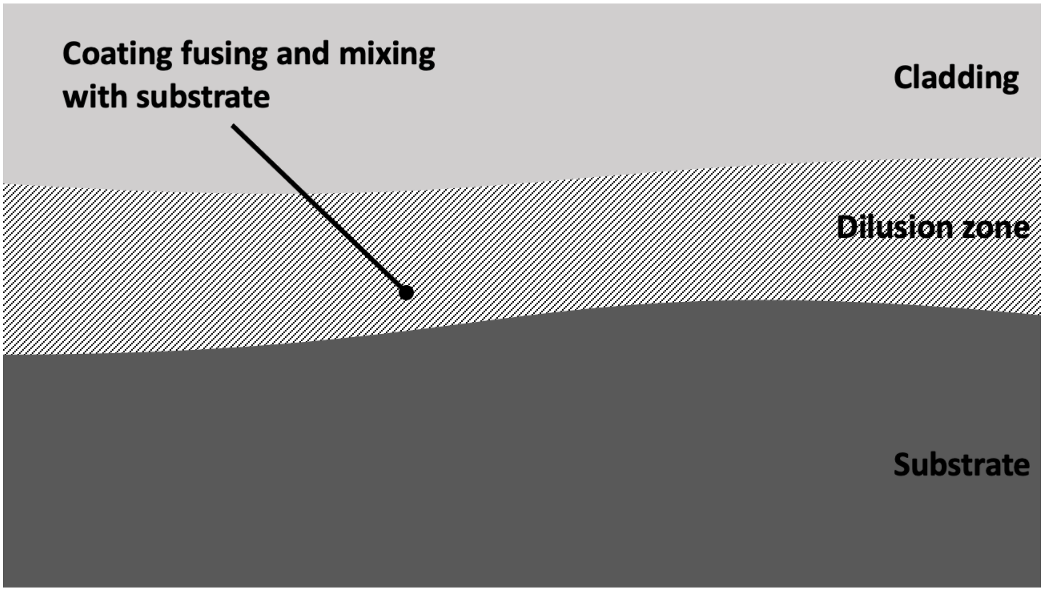

Figure 2.5: Schematic of metallurgical bonding structure.

2.8.2 Mechanical bonding

At mechanical bonding, there is no melting of the base material, and therefore dilution is not a problem in this case, which can be seen in figure 2.6. Instead, the bond between the substrate and the coating takes place by mechanical interlocking where the impacting particles are bonded with the substrate mostly through the impact of the incoming particles which solidify around the substrate surface irregularities[25], [26].

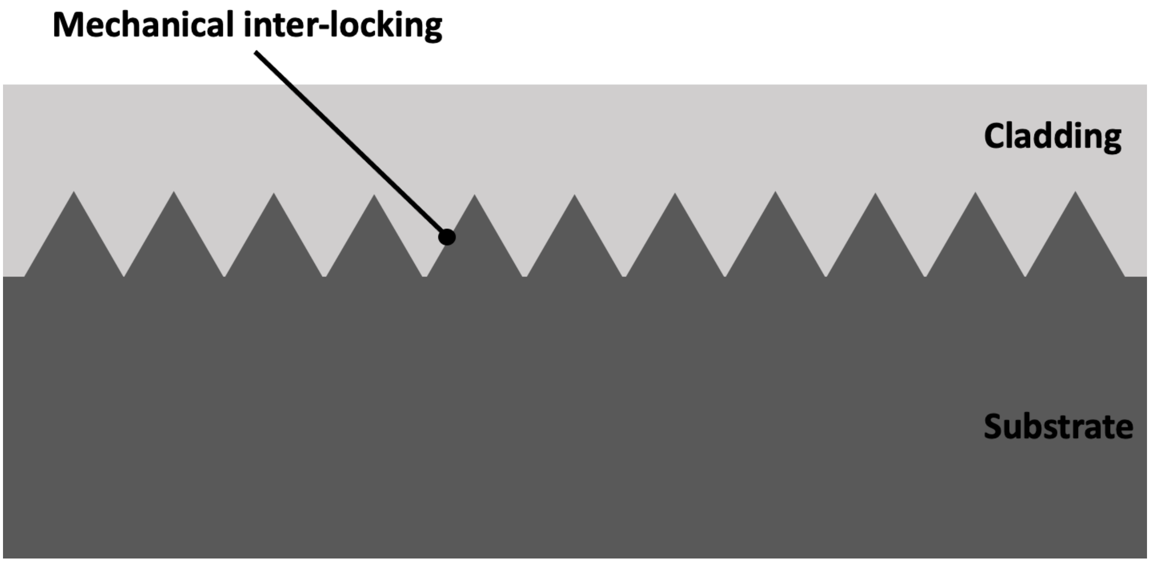

Figure 2.6: Schematic of mechanical bonding structure.

2.9 Coating materials

As explained in previous sections the cladding material must possess extraordinary properties to withstand aggressive and hazardous environments in which the oil- and gas industry consists of. The cladding material forms a thin layer and protects the underlying base material and which creates a durable and economical solution. Various materials can be used for the coating, based on the desired application. For laser cladding, materials such as Cobalt and Nickel-based alloys, Stainless Steel, Copper, Aluminium, Titanium, Bronze alloys, and certain composite materials are commonly used for improved erosion and corrosion properties. The same materials can be applied by thermal spraying as well as other materials such as ceramics and polymers. Various sources show that nickel alloys form good protection against corrosion and are well suitable for laser cladding and thermal spraying. In this thesis, three different materials were selected for deeper research. These are Hastelloy C-276, Inconel 625, and Incoloy 825.

2.9.1 Hastelloy C-276

Hastelloy C-276 is an solid solution strengthened austenitic superalloy consisting of elements shown in table 2.1.

Table 2.1: Composition of Hastelloy C-276 powder in wt%.

Cr	Mo	W	Fe	Mn	Ni	C	Si
15.5%	16%	4.5%	3%	1.2%	Bal.%	0.12%	0.5%

This composition of elements gives this material outstanding characteristics for aggressive environments such as oxidizing and reducing environments and is used in most industrial settings where aggressive substances are present and other materials fail. The combination of the high amounts of Molybdenum and Chromium and the addition of Tungsten makes the alloy very resistant to stress corrosion cracks, pitting corrosion, crevice corrosion, and general corrosion at high temperatures. The C276 alloy are proven to resist the corrosive effects of natural gas environments, which usually consists of hydrogen sulfides, carbon dioxide and chlorides. The alloy also exhibits resistance to corrosion by seawater, the marine environment and can operate in oxidizing atmospheres up to 1000°C. However, the alloy is also very expensive, partly because it is difficult to machine. The low carbon content of the alloy and the fact that it is resistant to carbide deposits during welding enables the alloy to be welded, which makes it an excellent cladding candidate[27].

In addition to the excellent corrosion properties, the mechanical properties of the alloy are also good. With a thermal expansion of $11.2\mu m/m$ between $20 - 100°C$ and an e-modulus of 205 GPa at room temperature, it has fairly similar properties as SS316L[28].

2.9.2 Inconel 625

Inconel 625 is a Nickel-based super alloy that possesses good strength properties, fabricability and outstanding corrosion resistance due to the containing elements chromium (Cr), molybdenum (Mo) and niobium (Nb). The chemical composition is shown in Table 2.2.

Table 2.2: Composition of Inconel 625 in wt%.

Cr	Mo	Mn	Fe	Co	Ni	Al	Nb + Ta
21-23%	8-10%	0.5%	5%	1%	58-71%	0.4%	3.2-3.8%

Inconel 625 can withstand high stress and a wide range of temperatures, both in and out of the water, as well as resist corrosion while being exposed to highly acidic environments. The high strength and superior corrosion resistance are derived from the stiffening effect of molybdenum and niobium on its nickel-chromium matrix. These excellent properties make Inconel 625 resistant against pitting and crevice corrosion, high corrosion-fatigue strength, high tensile strength, and resistance to chloride-ion stress-corrosion cracking. The high content of chromium makes this alloy very resistant to oxidizing environments, such as concentrated sulfuric acid, nitric acid, etc. In addition to the excellent corrosion properties, Inconel 625 also has good mechanical properties. With a melting temperature of $1300°C$ and a thermal expansion coefficient of $1.28 \cdot 10^{-5} 1/K$ at $20°C$, this alloy withstands a wide temperature range The superior fabricability of this alloy makes it weldable with most metallic materials, which makes it an strong candidate for a corrosion protection cladding[18], [29].

2.9.3 Incoloy 825

Incoloy 825 is a Nickel-iron-chromium superalloy with the addition of molybdenum, copper, and titanium. The detailed chemical composition is listed in table 2.3.

Table 2.3: Composition of Incoloy 825 in wt%.

Cr	Mo	Ti	Fe	Cu	Ni	Mn
19.5-23.5%	2.5-3.5%	0.6-1.2%	22%	1.5-3%	38-46%	1%

These additional elements, such as molybdenum and copper make this alloy resistant to reducing agents and acids. The addition of chromium makes it resistant to oxidizing conditions, such as nitric acid solutions. This means that the Incoloy 825 has reasonably good resistance to both reducing and oxidizing environments, but these are not outstanding as the C276 and Inconel 625 have. The alloy has good welding properties and is readily weldable by the normal processes, such as TIG, MIG, Laser, etc.[30].

2.10 Dilution

Dilution is something occurring in cladding or weld overlay processes where both cladding and some base material involve melting. The melting of the substrate is one of the major differences between the cladding and thermal spraying. This means that the final solidified coating consists of a mixture of the coating material and base material (figure 2.7). The composition of the coating is then defined by the percentage of dilution. This relation is shown in equation 2.1 where B is the amount of molten base material, and where $A + B$ is the sum of added coating material and the amount of molten base material.

$$Dilution = \frac{B}{(A+B)} \cdot 100\% \tag{2.1}$$

Dilution is an important factor when it comes to cladding and weld overlaying components. It does not only affect the composition and the microstructure of the cladding, but also the bonding strength between the deposited material and the substrate. Therefore it is important to have a good balance in the dilution. Low dilution leads to lacking of adhesion between different materials, and too high dilution will affect the surface material and may change its composition and properties such as wear and corrosion resistance[25].
A high rate of dilution due to high heat input have will increase the tendency of carbon diffusion when Nickel-alloy is welded to low alloy steel. This stimulates carbide formation at the surface layer, which can adversely affect the corrosion properties[31].

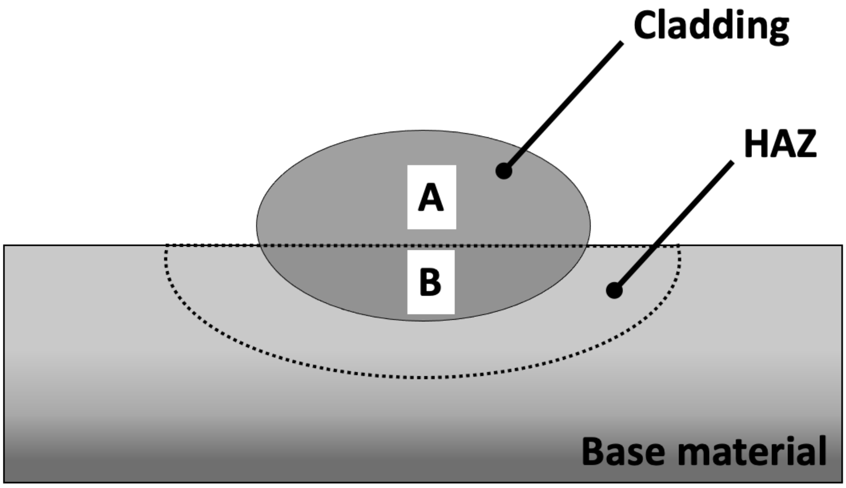

Figure 2.7: Dilution of cladding material.

3
Methods

This section describes the tests and measurements that were done in this study. Section 3.1 describe the material and process selection for the cladding. In section 3.2 the cladding process and the used equipment is presented. The test that was made on the clad flange is presented in section 3.3.

The cladding process was performed by an external company which is specialized in metal coating. Due to trade secrets according to the cladding company only the equipment in the cladding process is presented. Most of the tests to examine the properties of the C-276 cladding were performed at the Chalmers University of Technology, but some tests were also performed at the case company.

3.1 Selection process

A material selection was done to screen and rank possible materials for the cladding. The selection process started at the drawing board stage where all important factors affecting the product, such as the choice of materials and processing methods are considered.

3.1.1 Material selection

The material process started with a literature study to get an understanding of the material requirements, industrial requirements, and mechanisms of attack. After that Granta Edupack software Level 3 was used to make a rough material screening. Table 3.1 shows the selection criteria set in Granta Edupack.

Table 3.1: Selection criterias set in Granta Edupack.

Type	Value
Weldability	5
Sulfiric acid	Excellent
Crude oil	Excellent
Industrial Environment	Excellent
Marine Environment	Excellent

3.2 Cladding process and equipment

A suitable stainless steel flange that was big enough to cut out test rods was selected to be clad, the dimension of the selected flange is shown in figure 3.1. The flange was clad by laser powder cladding by a external company which are specialized on metal coating. The powder for the cladding was provided by Höganäs. The laser cladding included a cladding consisting of four layers to achieve a total thickness of 3mm after final machining. As can be seen in figure 3.2 the cladding process was performed by welding overlay C276 powder on the SS316L flange in a circular path.

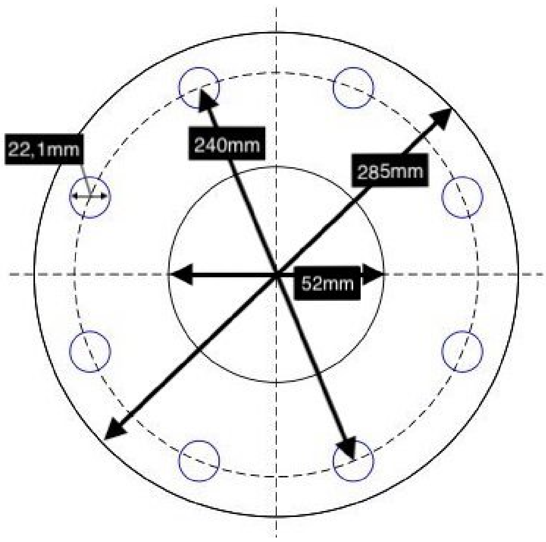

Figure 3.1: Dimensions of the selected flange.

The laser powder cladding were made with following equipment's:

- Robotic: ABB IRB4400
- Laser: IPG YLS 8000
- Powder feeder: Oerlikon Twin-150
- Optics: IPG D50

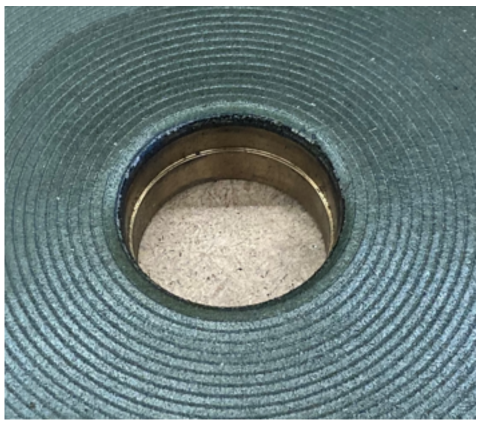

Figure 3.2: Laser powder clad flange before final machining.

3.3 Testing of clad flange

The tests were performed to examine the properties and the quality och the clad flange. The selection of the test was based on the standards ASME IX, ISO 15614-7, and API6A-2018 for coated materials. The test samples were cut from the coated flange using water cutting. A schematic of how the test samples was cut can be seen in Appendix 1.

3.3.1 Metallography and microscopy

In order to examine the microstructure of the clad flange, test samples were prepared perpendicularly to the cladding direction, from the middle of the total length of the test piece. The cross-section surfaces were prepared with:

1. Grinding and polishing to remove the saw marks and clean the surface.
2. The specimen surface was electrolytically etched for 30 seconds with Oxalic acid $C_2H_2O_4$ by 3V DC, and the clean with distilled water to stop the etching process to be able to distinguish between the two different materials and to examine the dilution and penetration depth of the clad layer and to detect any defects in the cladding.

The test specimen was examined with a stereo microscope (Zeiss axioscope 7) to detect any defects such as porosity, inclusions, lack of fusion, and hot cracks.

3.3.2 Hardness test

The hardness test was performed on the cross-section of the test specimen, which was etched with Oxalic acid. The measurements were performed by a Vickers hardness measurement device, with a measuring weight of 10kg. The impressions to measure the hardness was made according to figure 3.3.

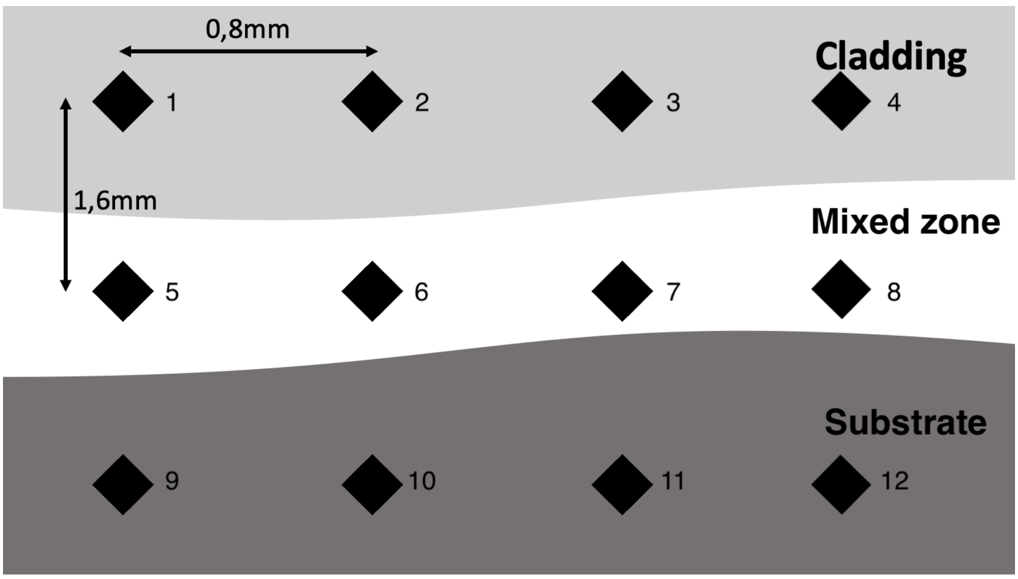

Figure 3.3: Positions of impressions for hardness test.

3.3.3 Penetration test

The penetration test was performed by a certified operator at the case company. Where the clad stainless steel flange face first was carefully cleaned by Bycotest C10 CleanerRemover to remove unwanted oil, greases, and contamination from the inspection area. The cleaned flange was air-dried for 5 minutes before the Bycotest RP20 Red Penetrant was applied by a brush and left for 20 minutes at 23 °C to be absorbed. After 20 minutes the penetrant was removed by Bycotest C10 CleanerRemover and air-dried for 5 minutes. After that Bycotest D30 Plus was sprayed over the clad flange face for crack indication.

3.3.4 Bend test

To control the adhesion between cladding and substrate a three-point bend test was performed. The bend test was carried out following the requirements of the standards ASME IX were two test samples with dimension 100x10mm was cut out from the flange. The test specimens were then bent at an angle of 180°, as can be seen, in figure 3.4.

Figure 3.4: Procedure of three point side bend test.

3.3.5 Chemical analysis

To be able to examine the chemical composition of the cladding layer an SEM/EDS analysis on the cross-section of the test sample was carried out. The chemical analysis was measured on two places at the cross-section. In connection with the EDS analysis, the cross-section was also examined by SEM to detect any defects in the interface region such as porosity, inclusions, lack of fusion, hot cracks, and also to measure the thickness of the cladding layer. To perform this analysis the sample preparation was made by grinding and polishing the test samples which were cut out from the clad flange.

4
Results

The results are divided into three main parts:
1. The results of the material selection studies. With a focus on corrosion resistance cladding
2. The results of the process selection. With a focus on cladding processes.
3. The results of the performed tests of the chosen cladding, with a focus on properties and quality.

4.1 Material selection of cladding

The initial material selection process was made in Granta Edupack where three different alloys were chosen as possible candidates for the cladding based on the set constraints presented in table 3.1. The following materials were found to meet these requirements and were chosen to study in more detail, Hastelloy C276, Inconel 625, and Incoloy 825. Table 4.1 presents some differences between the alloys in important parameters.

Table 4.1: Properties of possible cladding materials for oil and gas industry.

Materials	Types of resistance	H_2S	CO_2	Service temperature
C276	Reduction resistance	Excellent	Excellent	-273 - 1010°C
Inconel 625	Oxidation resistance	Excellent	Excellent	-273 - 980°C
Incoloy 825	Oxidation/Reduction resistance	Moderate	Moderate	-273 - 540°C

As can be seen from the table above there is no big difference between the materials when it comes to important parameters for the specific application and industry. The C276 alloy seems to be most suitable as a cladding material as protection for stainless steel flanges due to its excellent resistance to reducing corrosion and its wide range of service temperatures. This material selection will be further discussed in section 5.2.1.

4.2 Process selection of cladding

The process selection was performed by studying the most common methods of cladding where important parameters such as availability, heat input and bonding type were in focus. Table 4.2 presents the selected methods with the best potential

Table 4.2: Chemical composition (wt.%) at 1.5 mm from the surface.

	Laser cladding	CMT	GTAW	Thermal spraying (HVOF)
Temperature	$\approx 500°C$	$600 - 800°C$	$> 3000°C$	$2000°C$
Thickness	0.5-2mm	0.5-2mm	0.5-2mm	0.1-0.3mm
Bonding	Metalurgical	Metalurgical	Metalurgical	Mechanical

to produce a high-quality cladding for corrosion protection.
As can be seen in table 4.2, three of the methods are quite similar when it comes to thickness and bonding type. Taking all parameters into the aspect, laser cladding seems to be the most suitable method to produce a corrosion protection cladding due to its strong bonding to the substrate and low heat input.

4.3 The results of the laser cladding testing

The laser cladding studies started with a general inspection by the naked eye of the powder-clad flange, which was clad by 4 layers. No defects or cracks were found on the cladding surface. The other laser cladding test results are divided into the following sections:

1. Penetration test: surface defects and density of cladding
2. Bend test: adhesion between cladding and substrate
3. Micro/macro examination: porosity and micro cracks
4. Hardness
5. Chemical composition: level of dilution

4.3.1 Penetration test

In order to examine the surface quality and check how dense it is, a penetration test was done on the cladding surface. Figure 4.1 shows the result of the penetration test. As can be seen in figure 4.1, some linear cracks were found around the center hole of the flange. Apart from that, the remaining surface does not tend to have any defects. Due to the linear cracks around the center hole the cladding did not pass the penetration test.

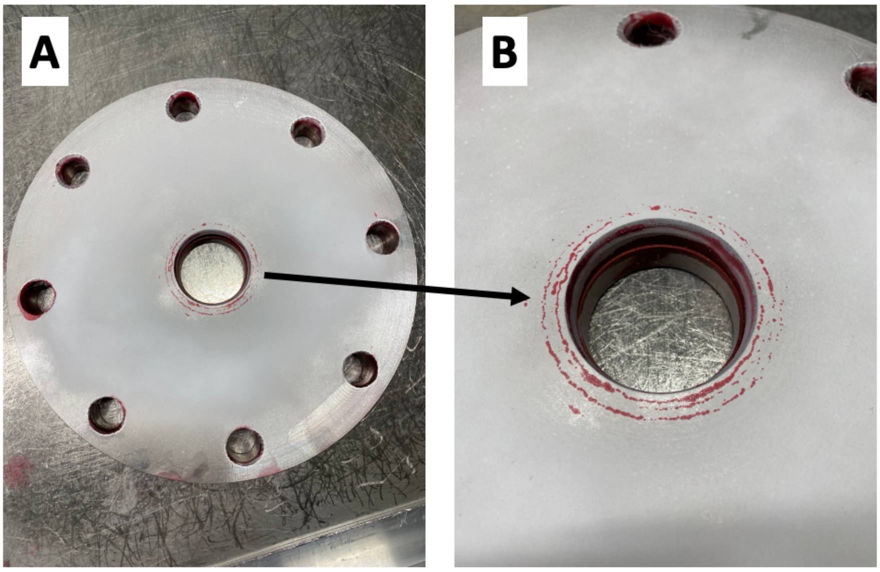

Figure 4.1: Results form the performed penetration test a) overview of the hole clad surface b) indicating of crack and defects on the surface.

4.3.2 Bend test

To check how well the adhesion between the cladding and the substrate is, a bend test according to ASME IX was performed. Figure 4.2 shows the specimens after the bending test. As can be seen in the figure below the substrate material stayed intact during the bending. The cladding instead failed to remain intact during bending which resulted in cracks along the weld strings. However, there are no indications of any separation in the interface between the coating and the substrate which indicates a good adhesion.

Figure 4.2: Results form the performed bend test on two test samples, indicating the adhesion between cladding and substrate.

4.3.3 Micro/macro examination

Microscopic analysis was performed on the cross-section to examine the quality of the clad test sample. As can be seen in figure 4.3, the cross-section consists of three regions the substrate, the mixed zone, and the cladding layer. As also can be seen in the figure below no defects are observed in the substrate material or the interface between the two materials. The defects start at the mixed zone in form of pores and hot cracks and continue through the cladding layer. The biggest crack is $\approx$ 1mm long. The total clad layer was measured to $\approx$ 2.8mm and the mixed zone was measured to $\approx$ 0.9mm

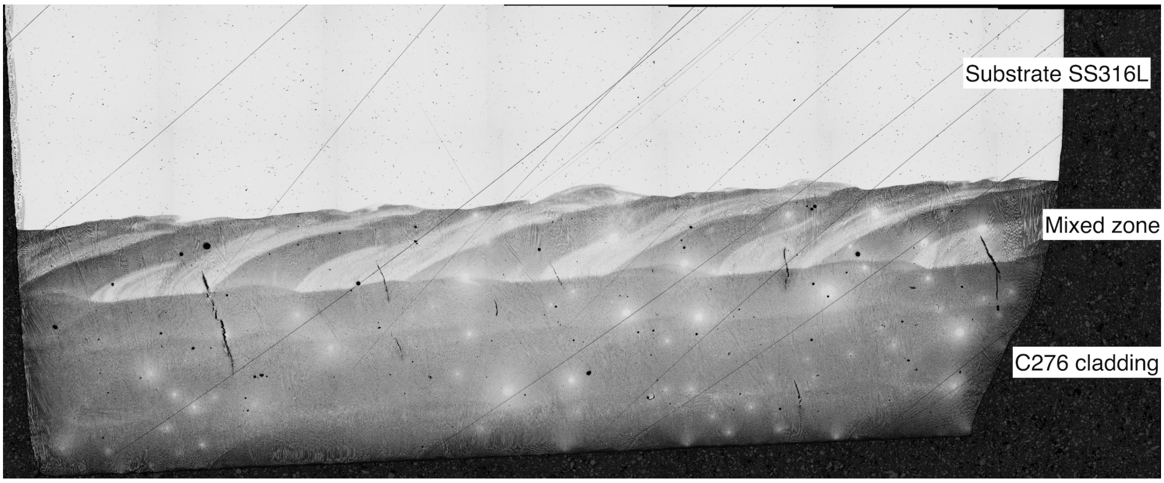

Figure 4.3: Interface between substrate and clad layer.

4.3.4 Hardness test

From the hardness testing on the clad test sample, the Vickers hardness values were calculated by equation 4.1.

$$HV = 1.854\frac{F}{D^2} \tag{4.1}$$

Where F is set to 10 kgf and D is the mean of the two indent diagonals. Table 4.3 presents the hardness values at different positions on the cross-section of the clad test sample. Table 4.3 presents the measured hardness values at different positions of the cross-section, where indent nr. 1, 2, 3, and 4 are at the clad surface, indent nr. 5, 6, 7 and 8 are on the mixed zone between the two materials and indent nr. 9, 10, 11 and 12 are on the substrate material. The corresponding positions to the hardness values can also be seen in figure 3.3. The mean values of the hardness at the different position of the cross-section shows that the cladding layer is hardest followed by the dilution zone. The softest part of the cross-section is the substrate material. As also can be seen from table 4.3.

Table 4.3: Hardness values of different positions of the cross-section.

Indent nr	1	2	3	4	5	6	7	8	9	10	11	12
Hardness value	258	248	253	264	252	256	247	234	185.8	185.8	180	185.8

4.3.5 Chemical analysis

In order to maintain the excellent properties of the C276 alloy, no major changes in chemical composition must occur during the manufacturing process. Tables 4.4 and 4.5 show the chemical composition at 0.75mm and 1.5 mm from the surface of the cladding.

Table 4.4: Chemical composition (wt.%) at 0.75 mm from the surface.

Cr	Mn	Fe	Co	Ni	Mo	W
14.86%	0.24%	2.29%	1.55%	62.33%	14.21%	4.51%

Table 4.5: Chemical composition (wt.%) at 1.5 mm from the surface.

Cr	Mn	Fe	Co	Ni	Mo	W
14.68%	0.30%	4.07%	1.58%	62.46%	12.92%	3.98%

The results presented in Tables 4.4 and 4.5 show a very low Fe content at the surface and in the middle of the cladding, which indicates a low dilution during the welding process.

5

Discussion

In this chapter, the discussion of the obtained results is presented, this chapter is divided into three parts associated with the research questions. The chapter ends with suggestions for future research.

5.1 Research Question 1

What requirements are set by the industry regarding corrosion protection with coating?

The main reason for using coatings is to protect the underlying material and thereby increase the utilization and service life of the specific component. The oil and gas industry is one of many industries that use this method to avoid corrosion and wear and tear which is the most common problem in the industry, thus avoiding catastrophic consequences. Since this is an industry that handles toxic substances for both the population and nature, the industry is regulated by many different standards to ensure safe and sustainable production. When reviewing the standards for corrosion protection coating in the oil and gas industry (NACE MR0175, ASME IX, API6A-2018, and Norsok M 630), there were two clear requirements, the thickness of a corrosion resistance coating must be at least 3 mm after final machining. As well as how a corrosion protection coating should be tested to meet the requirements on mechanical properties. In addition, the standards also list the risks that should be considered for use in upstream oilfield equipment where sulfide stress corrosion cracking and crevice corrosion are common in acidic environments such as oil/ gas/saltwater mixtures where H_S and CO_2 are present. Apart from that, no more clear criteria are found on metal coating for corrosion protection.

A large proportion of these standards treat plastic coatings, such as dip coating and painting of equipment and pipelines to prevent corrosion. This shows that this is a more advantageous method when it comes to cost and availability and therefore a more common method compared to metal coating in the current situation. But with the large investment in metal coating development where both new materials and methods are developed, this will become an increasingly common method for corrosion protection.

5.2 Research Question 2

Which materials and coating methods are best suited for corrosion protection? As coatings are a very common solution for corrosion and wear protection, there are many different materials and methods to use today. After determining the requirements of the industry that the coating must meet which are:

- have excellent corrosion resistance against aggressive substances such as CO_2, H_2S in a marine and industrial environment.
- be weldable with the probe which connects the tank with the measuring system.
- the coating process have to be conventional

Many of these materials and methods were sorted out and where metal cladding was considered to be the best solution to meet the set requirements.

5.2.1 Material selection

The material selection process started with a screening of competitors to see if anyone else has coated flanges to prevent corrosion. When reviewing competitors, no solutions were found that included metal coating of flanges. Most had similar solutions as the case company. Subsequently, a material selection was made in Granta Edupack where three materials were considered to meet the set requirements for corrosion and weldability. Three different types of nickel alloys were selected and were considered to best meet the set requirements, see Table 4.1. All three materials are very common in the oil and gas industry and have similar properties when it comes to corrosion resistance, but of these three, the Incoloy 825 has the worst corrosion resistance in both reducing and oxidizing environments. Incoloy 825 also has the least temperature range, which gives the other materials an advantage. What differentiates the Hastelloy 276 and Inconel 625 is the type of corrosion type they handle best, with the Hastelloy C276 performing best in reducing environments and the Inconel 625 in oxidizing environments. In the oil and gas industry, carbon dioxide (CO_2) and hydrogen sulfide (H_2S) are commonly present, and water is their catalyst for corrosion. The most common type of corrosion is a combination of these two gases which results in molecules being released into the electrolyte. In this case, Hastelloy C276 has more molybdenum content than alloy 625, which has a very good anti-reduction effect.

5.2.2 Process selection

When choosing a method for applying the cladding to the stainless steel flange, four different methods were studied that were considered to be able to do this. All these methods are common in corrosion protection claddings and perform differently depending on the application.

Thermal spraying is the method that gives the thinnest possible cladding of all these, which is an advantage when tight tolerances are important. With thermal spraying, complex geometries can also be coated with a wide range of coating materials, such as metal, alloy, ceramic, plastic, and polymer. However, this method only allows a mechanical bonding between the cladding and the substrate which is not so strong. This, together with the fact that the cladding is weldable, makes this cladding method unsuitable for this purpose.

GTAW is a cladding method that produces high-quality and smooth welds. And leaves a very strong metallurgical bond between the different materials. However, this process is performed at a very high temperature, which in turn provides a high heat input. This entails a high degree of dilution between the different materials, which in turn can affect the final properties of the cladding material.

The two remaining cladding methods give relatively similar results with a strong metallurgical bond between the different materials without generating a high heat input. This means that both of these methods have a good potential to generate good corrosion protection cladding. What decided that laser cladding was finally chosen was that the method is more common and more readily available.

5.3 Research Question 3

How does the selected coating perform in the test requirements set by the industry? To evaluate the results of the developed prototype, a number of tests were performed. These tests were performed according to the ASME IX standard to validate the generated cladding. The results of these tests are discussed in the following sections.

5.3.1 Penetration test

Penetration testing was performed to examine defects in the surface of the coating. The result of the test showed that there were linear cracks around the center hole of the flange, which failed it in this test. These indications may be due to incorrect processing of the cladding after the welding, as these cracks appeared near the edge of the center hole. The remaining part of the clad surface showed no signs of cracks or other defects.

5.3.2 Bend Test

The side bend test of the test samples from the coated flange was performed to check the adhesion between the substrate and the cladding. The results from the bending test show that the cladding still remains on the substrate and the cladding showed no signs of separating from the substrate. From the result, it can also be seen that cracks have formed after the bending. These cracks clearly appear to have arisen between the weld strings, which may mean that there has been a poor overlap between the weld strings. These cracks can also be due to internal stresses in the material that have arisen during the welding process. From the results of the hardness test we can also see that the C276 cladding has a significantly higher hardness compared to the SS316L substrate. This can lead to the cladding being more brittle, which can be a reason for the crack formation during bending. However, it should be suggested that this bending test was performed in the most extreme cases within the given standards. When the test rods were fabricated within the smallest possible dimensions to perform this test due to the size of the flange, the test had to be done with a small radius which make it extreme.

5.3.3 Micro/macro analysis

To ensure that a coating lasts for a long time, it is important that it does not contain too many defects such as hot cracks, porosity, and inclusions. Therefore, the cross-section of the clad test bar was examined with a microscope. From the microscopy analysis of the cross-section, one can observe different types of defects in the cladding. Pores spread over the cladding and the mixed zone can be observed, however, porosity is not considered to be a major threat to welding strength or fatigue. According to the ISO 15614-7 standard for weld overlay, pores as large as 2 mm in diameter are acceptable in weld overlay. The second type of defect that can be observed on the cross-section is hot cracks, which appear to be solidification cracks. It is difficult to say why these occur it can either be due to alloy composition or welding parameters. Previous studies[32] have shown that small amounts of impurity elements, such as Sulfur (S) and Phosphorus (P) can have a contributing effect to hot cracking. Even small amounts of Boron (B) can have major effects on weldability and be a contributing factor to hot cracking. As this work did not focus on the laser cladding process, it is difficult to say whether the welding parameters have an impact on its defects.

5.3.4 Chemical analysis

The chemical composition is a very important parameter that gives the material its properties. The results from the chemical analysis at the surface showed that the chemical composition of the cladding did not change significantly. Even in the middle of the cladding, no major differences in the chemical composition are noticed compared to the original ones. The Fe content was well below 5 wt.% Which was the goal to ensure continued good corrosion protection. The fact that the chemical composition of the cladding did not change much indicates that there was no major mixing during welding.

5.4 Further work

As this work was limited to making only one material selection and manufacturing process of a corrosion protection coating for flanges in the oil and gas industry, no account was taken of how the actual cladding process was performed. This means that no consideration was given to parameters used during the manufacturing process, such as welding speed, feed rate, pre-heating, power, etc. To achieve an even better coating, these parameters in laser cladding should be studied to find the best possible settings for this application.

This study also limited itself to the current construction of the flange, which limited the number of coating processes, and entailed requirements such as weldability of the coating. In the case of a redesign of the flange, several possibilities of corrosion protection such as plating could be made possible.

Finally, it would be interesting to test the developed prototype against corrosion to see how it performs, this was unfortunately not included in this work due to the limited time frame.

6 Conclusion

This report sought to investigate the possibilities to protect a stainless steel flange from corrosion by cladding. A material- and process selection was done and the nickel alloy C276 applied by laser powder cladding was considered to have the best potential and meet the requirements set by the industry. Based on the material- and process selection a prototype was developed and tested to validate the properties.

From the various tests of the developed prototype, the author found that there is a good potential to achieve very resistant corrosion protection by coating with nickel alloy C276, with a great adhesion with the substrate. Through further optimizations of the various welding parameters, there is a good potential to achieve a defect-free and resistant coating.

This early study provides a good basis for further development to develop alternative corrosion protection to today's solutions. However, more work is needed to achieve a defect-free coating and to test it in a real environment to see how it perform.

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Appendix

A.1 Appendix 1

Schematic of how the test samples was cut from the clad flange.

Figure A.1: Schematic of sample cut from clad flange

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