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Faculty Of Mechanical Engineering

Department Of Material Science



Diploma Thesis

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According to Act No. 111/1998 Sb. on Higher Education Institutions you have received diploma work with theme:

Anticorrosion protection of pipeline

Instructions for elaboration:

(List principal goals and recommended methods for elaboration of the master thesis)

1. Familiarize yourself with issue of corrosion and anticorrosion protection of pipeline systems.
2. Specialize in protection methods using coatings and methods of their application.
3. Elaborate a critical survey about testing methodologies of barrier coatings.
4. Investigate corrosion resistance of selected systems coating-substrate.
5. Evaluate your results and formulate conclusions.

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Elaboration form of the diploma work:

- report with extent from 30 to 40 pages
- attachments and/or appendices

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- [1] ASM Handbook - Volume 13 , Corrosion. 7th Edition. ASM International: Materials Park - Ohio, 2001. 1415 p. ISBN 0-87170-019-0
- [2] ASM Handbook - Volume 5 , Surface Engineering. 1st Edition. ASM International: Materials Park - Ohio, 1999. 1039 p. ISBN 0-87170-384-X
- [3] Journal Articles from Elsevier Publisher

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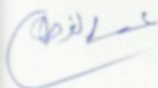
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Thanks

I want to give especial thanks to how was supervisor on my Diploma work to, **Ing. Vladimír Nosek** and to the head department of material science, **Prof. RNDr. Petr SPATENKA, CSc.** and also especial thanks for the dean of faculty of mechanical engineering , **doc. Ing. Petr LOUDA, CSc.**

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Annotation

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This diploma thesis presents the discussion of the problems in pipeline and cost and losses of corrosion in addition of that we will have information about characteristics of environment effect on internal and external pipeline and the methods of protection against corrosion by using many types of coatings, and we will discuss also methods of testing and monitoring of corrosion. For the experimental part we will examine some types of coatings applied to carbon steels like TiO_x , TiN_2 , paint coating (zincethylsilicate) and some types of polyethylene powder, by using of porosity test and sulfur dioxide test with general condensation of moisture.



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Annotation

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Tato diplomová práce diskutuje o problémech týkajících se potrubí a finančních ztrát díky korozi, navíc také podává informace o charakteristice vlivu prostředí na vnitřní a vnější potrubí a metody ochrany proti korozi užitím mnoha druhů povlaků, a také diskutuje o metodách testování a monitorování koroze. V pokusné části zkouší některé typy povlaků aplikovaných na uhlíkových ocelích jako je TiO_2 , TiN , nátěrové povlaky (zincethylsilicate) a některé typy polyethylenového prášku s použitím zkoušek pórovitosti a zkoušky s oxidem siřičitým s povšechnou kondenzací vlhkosti.

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List of symbols and abbreviations

D.C.....	Direct current.
FHWA.....	Federal high way administration.
FBE.....	Fusion bounded epoxy.
HDPE.....	High density polyethylene.
HT.....	High temperature.
HP.....	High pressure.
LCC.....	Life cycle costing.
LDPE.....	Low density polyethylene.
MIC.....	Microbiologically influenced corrosion.
MDPE.....	Medium density polyethylene.
PE.....	Polyethylene.
PASS.....	Paint adhesion on ascribed surface.
ROV.....	Remotely operated vehicle.
SCC.....	Stress corrosion cracking.
SRB.....	Soleplate reducing bacteria.
SSCC.....	Sulphide stress corrosion cracking.
SSR.....	Slow strain rate.
TW.....	Trailing wire.
UV.....	Ultra violet.

Introduction

Corrosion specifically refers to any process involving the deterioration or degradation of metal components. The best known case is that of the rusting of steel. Corrosion processes are usually electrochemical in nature, having the essential features of a battery. When metal atoms are exposed to an environment containing water molecules they can give up electrons, becoming themselves positively charged ions provided an electrical circuit can be completed. This effect can be concentrated locally to form a pit or, some times, crack, or it can extend across a wide area to produce general wastage. Localized corrosion that leads to pitting may provide sites for fatigue initiation and, additionally, corrosive agents like seawater may lead to greatly enhanced growth of the fatigue crack. Pitting corrosion also occurs much faster in areas where micro structural changes have occurred due to welding operations.

Corrosion and cracking on the external or internal surfaces of in-service pipes, tanks, or other industrial assets reduces the integrity of the material and potentially reduces the service life of the equipment. Pipeline plays an extremely important role throughout the world as a means of transporting gases and liquids over long distances from their sources to the ultimate consumers. Degradation of pipelines is the result of the persistent attack by the environment on pipeline materials (coatings, welds, pipe, etc.). Buried pipelines are located within ever changing environmental conditions that may lead to a corrosive environment.

This theme of diploma present the discussion of the problems in pipeline and cost and losses of corrosion in addition of that I will have information about characteristics of environment effect on internal and external pipeline and the methods of protection against corrosion by using many types of coatings, and I will discuss also methods of testing and monitoring of corrosion. For the experimental part I will examine some types of coatings applied to low carbon steel like TiO_x , TiN_2 , paint coating (zincethylsilicate) and some types of polyethylene powder, by using of porosity test and humidity chamber test.

2. Theoretical part

2.1 Types of corrosion of pipeline.

2.1.1 Typical corrosion types on pipelines

Uniform or general corrosion: Is defined as corrosion that occurs at substantially the same rate over the entire exposed surface of a metal. When uniform corrosion occurs, very small electrochemical cells are established on the surface of the metal due to small differences in metal composition or the non uniform nature of corrosion product layers that form on the surfaces. Corrosion of sheet piling takes the form of both uniform roughening of the surfaces and pitting. Distributed loss of metal is of more interest, since this contributes to a reduction in the strength of the section and potential failure by overstressing. Corrosion resulting from exposure to the atmosphere and fresh water immersion is usually of the uniform type. [1]

Crevice corrosion: The crevice corrosion is created as a narrow opening or spaces (gaps) between metal to metal or non metal to metal components. Example: Corrosion of stainless steels at spots where two surfaces are pressed together, e.g. steel to plastic, or to the same steel (bolts and nuts). Caused by loss of passivity in the resulting water film at these crevice by a differential concentration cell.

Crevice corrosion can initiate by depletion of inhibitor in the crevice, a shift to acid conditions in the crevice, a build-up of aggressive ion species in the crevice, and depletion of oxygen in the crevice. Preventing of crevice corrosion can be created by the following: Geometrical (type of crevice :metal to metal, non metal to metal, crevice gap, crevice depth, exterior to interior surface area ratio) Environmental (bulk solution: oxygen content, pH, chloride level, temperature, agitation, mass transfer, diffusion and convection). Electrochemical reaction (metal dissolution: oxygen reduction, hydrogen evolution). Metallurgical (alloy composition: major elements, minor elements, impurities, passive film characteristics) [2]

Erosion corrosion: Is acceleration in the rate of corrosion attack in metal due to the relative motion of a corrosive fluid and a metal surface. Erosion is initiated by:

The increased turbulence caused by pitting on the internal surfaces of a tube can result in rapidly increasing erosion rates.

Erosion corrosion can also be aggravated by faulty work man ship. For example, burrs left at cut tube ends can upset smooth water flow, because localized turbulence and high flow velocities, causing high pitting rates. Erosion can be controlled by removing solids from the stream and by mechanical design [2]

Pitting corrosion: Is a localized form of corrosion by which cavities or "holes" are produced in the material. Pitting is considered to be more dangerous than uniform corrosion against. Corrosion products often cover the pits. A small, narrow pit with minimal overall metal loss can lead to the failure of an entire engineering system. Pitting corrosion, which e.g. is almost a common denominator of all types of localized corrosion attack.

Pitting is initiated by:

- Localized chemical or mechanical damage to the protective oxide film.
- Water chemistry factors which can cause breakdown of a passive film are acidity, for example (low dissolved oxygen concentrations and high concentrations of chloride)
- Localized damage to, or poor application of, a protective coating.
- The presence of non-uniformities in the metal structure of the component, nonmetallic inclusions.

Pitting can be prevented by the following:

- Reducing the aggressivity of the environmental,
- Upgrading construction materials, and modify the design of the system. [2]

Environment-induced cracking: It is result from the joint action of mechanical stresses and corrosion. Stress Corrosion Cracking (SCC) falls within this group. This type of corrosion mechanism and its rate of attack depend on the exact nature of the environment (air, soil, water, seawater) in which the corrosion takes place. In today's industrial setting, the waste products of various chemical and manufacturing processes find their ways into the air and waterways. Many of these substances, often present only in minute amounts, act as either catalysts or inhibitors of the corrosion process. The corrosion engineer then needs to be on the alert for the effects of these contaminants. [3]

Microbiological corrosion: Microbes (bacteria) can cause corrosion, even on stainless steels. The bacteria do not directly eat the metal, but their waste products are corrosive. They also can cause the development of differential concentration cells, leading to pitting. The most common type is the *Soleplate* reducing bacteria or SRB's, which convert sulphates to sulphides and H_2S . Counter measures are not giving them anything to eat (sulphates, nitrates, sources for carbon which they also need), and killing them by injection of bactericides or chlorination. This is difficult because they hide behind slime deposits which they create. [4]

H_2S corrosion: H_2S can cause pitting corrosion on several alloys. In most cases a protective sulphide is formed; weak spots in this layer can lead to enhanced corrosion. With ferrite steels, dissolved H_2S causes H-atoms to diffuse into the metal. The reason for this is that sulphides interfere with the reaction of H-atoms, originating from corrosion reactions, to H_2 molecules. This causes the metal to become brittle. There are two forms of damage:

Sulphide stress corrosion cracking or (SSCC), and hydrogen induced cracking or HIC.

H_2S corrosion can be initiated by the following:

- High pressure H_2 gas bubbles which grow at intermetallic.
- Impurities like manganese sulphide stringers. Stepwise cracking.

[5]

Galvanic corrosion: This is an aggressive and localized form of corrosion due to the electrochemical reaction generally between two or more dissimilar metals in a conductive environment. The more electro-negative material (the anode) is attacked by the more electropositive material (the cathode). The most common example of such corrosion activity, widely found throughout heavy accumulations operations, is the direct connection of brass valves to carbon steel pipe, or between copper and steel pipe. [2]

2.1.2 Types of internal and external pipeline failure modes.

General external corrosion: it's metal loss due to electrochemical, galvanic, microbiological, or other attack on the pipe due to environmental conditions surrounding the pipe.

General internal corrosion: it's metal loss due to chemical or other attack on the steel from liquids on the inside of the pipe. Electrochemical attack can also occur in local cells, but this condition is less frequent.

Pipeline failure modes: Pipeline has several potential failure modes or threats, including internal and external corrosion:

The main external corrosion failure modes are:

- Sea water corrosion, scowering, abrasion of the coating, and sea bottom movement.
- Galvanic corrosion (dissimilar metals in an electrolyte).
- Oxygen concentrations cell corrosion (pitting and crevice corrosion)

The main internal corrosion failure modes are:

- Acid gasses and organic acids combine with water.
- Erosion, and erosion-corrosion caused by sand and entrained particles (or droplets).
- Scaling caused by incompatible fluids. [6]

2.2 Corrosion costs for pipeline.

2.2.1 Costs for general corrosion.

Corrosion costs in the United State the industry, government, agencies an estimated 276 billion/year that was according to the study supplied in 2001 by cc technologies for the federal highway administration (FHWA) responses to the study were reviewed from the group base, the United State, United Kingdom, Saudi Arabia, Canada, Argentina, India, Japan, South Africa, Venezuela, and China and the major interest was in the following industries:

Paints, coatings and lining, plating and surface finishing, powder coatings, plastics, composites, flow control, water conditioning, water distribution, heating, fabrication, machinery, product finishing, pipeline. The cost of corrosion can be defined in different ways depending on what is included and who is affected.

The cost of corrosion is defined as the corrosion fraction of design, manufacturing, operation and maintenance, technology development, and assets value loss. While emphasis is placed on current corrosion cost, in some cases, changes in the cost of corrosion could be addressed by examining changes in corer-onion control practices.

There are three important concepts that are nearly used in the analysis of the result of the corrosion cost and they:

- Corrosion management and it's includes all activities like design, manufacturing, maintenance, inspection, repair, rehabilitation and removals through the lifetime of structure, that are performed to prevent corrosion, repair its damage and replace its structure.
- Life cycle costing (l.c.c): It is cost of all cash expenditures to the end of the structures life, including construction cost, the cost of maintenance and the cost of outages.
- Cost benefit analysis : Its have benefit of spending money on corrosion issues in some causes prevention of corrosion failures is cost too much where a corrosion failure may have minimal impact and it can replacing apart at low cost. And it's the most economical solution.

The annual cost of corrosion can be divided in two groups, direct costs and indirect costs.

The direct costs of corrosion have two main components:

- The cost of design, manufacturing, and construction, examples: material selection (such as stain less steel and replace

carbon steel), an additional material, such as increased wall thickness for corrosion allowance material used to mitigate or prevent corrosion, such as coating, sealing, corrosion inhibitors and cathodic protection.

An application including the cost of labor and equipment.

- The cost of management:

Corrosion-related inspection..

Corrosion -related maintenance.

Creepers due to corrosion.

Replacement corroded part.

Inventory of backup components.

Rehabilitation.

Loss of productive time.

The indirect costs such as the traffic delay due to the bridge repairs and rehabilitation that are more difficult to turn over to owner or operator of the structure. these costs can be measured and determined by risk-based analyses.[7]

2.2.2 Costs for pipeline corrosion.

Corrosion of transmission pipelines costs the United States every year from 5.4 to 8.6 billion dollar according to NACE federal study.

The last amount of cost can be divided in to three parts:

- The cost of failure.
- Capital.
- Operation and maintenance (O&Q)

The last three part of costs of corrosion is based on 1998 costs and doesn't content the new regulations related to pipeline inspection.

Although data management, system quantification through the use of global positioning surveys, remote monitoring, and electronic equipment developments have provided significant improvement in several areas of pipeline corrosion maintenance and monitoring.

The last amount of corrosion costs just includes the direct costs owner/ operator costs) and does not includes indirect costs (costs to the user or public), where indirect costs estimated to be similar to direct costs.

The report of corrosion costs divided into five sector categories, which were sub divided into 26 industrials sectors, and the transmission pipelines is one of the 26 sectors.

Corrosion is the primary reason for aging and deterioration of a buried ` pipeline, so the cost of corrosion prevention is directly related to

preservation of valuable asset. The major impact of corrosion on transmission pipelines is economic, safety and environmental. Economics of the major impact of the corrosion is affected in the cost to preserve major assets. [8]

The current study showed that technological changes have provided many new ways to prevent corrosion and the improved use of available corrosion management techniques. However, better corrosion management can be achieved using preventive strategies in non-technical and technical areas.

These preventive strategies include:

- Increase awareness of large corrosion costs and potential savings.
- Change the misconception that nothing can be done about corrosion.
- Change policies, regulations, standards, and management practices to increase corrosion savings through sound corrosion management.
- Improve education and training of staff in recognition of corrosion control.
- Advance design practices for better corrosion management.
- Advance life prediction and performance assessment methods.
- Advance corrosion technology through research, development, and implementation. [9]

2.3 Characteristics of corrosion environment.

2.3.1 Environmental effects on corrosion.

Corrosion is reaction between a metal or alloy and its environment. Corrosion is effected by the properties of both the metal or alloy and the environment.

The environment variables are in the following:

- pH (acidity)
- Oxidizing power (potential)
- Temperature (heat transfer)
- Velocity (fluid flow)
- Concentration (solution constituents)

pH (acidity): Is the actual amount of acid as defined by the hydrogen ion concentration. The importance of the hydrogen ion lies in its ability to interact with an alloy surface. Many of commercially alloy interest form an oxidized surface region, the outer most atomic layer of which often contains hydroxide like species when water is present. Such structure would tend to have a dependence on hydrogen ion concentration, possibly through a reaction that can be one step in corrosion.

Oxidizing power: Is effected because it has ability to remove or add electrons from the metals so as to oxidize or reduce to surface. For practical applications such as increasing passivity by altering the surface oxides (anodic protection) or preventing corrosion by supplying electrons to the metals that will be normally yielded by metal corrosion (cathodic protection), where the anodic reaction rate is shifted or changed in the protected metal. The alteration of the surface state to impart passivity is normally accomplished by anodic polarization of the metal or alloy surface to potential noble to the corrosion potential. The addition of constituents to the environment may alter the surface potential to create a passive film. In this case the constituent reacts with the metal to form a tenacious metal metal–oxide compound that passiveness the surfaces. Polarization of the surface potential in the active or cathodic direction can also be used to decrease corrosion. When the potential is lowered by means of an external voltage source, the technique is known as the cathodic protection.

Temperature: Is complex external variable, so it's analogous to potential obviously and potential difference creates a current flow the objective of which is to eliminate the potential differences.

Temperature can effect corrosion in a number of ways:

- The corrosion rate increases exponentially with an increase in temperature if the corrosion rate is governed completely by the elementary process.
- If the constituent in the solution has limited solubility a temperature change can alter the concentration of the constituent, which this alteration can have profound effect on corrosion.
- For the closed system as the temperature increases the oxygen will tend to leave the liquid but it cant escape from the vapor spaces above the liquid, when the temperature increase the vapor pressure also increase and then it tends to maintain the oxygen concentration in the liquid and then the corrosion rate continue to increase with temperature because of effect temperature with viscosity, diffusivity.
- For the open systems oxygen can escape from the immediate vicinity of the liquid, so the vapor pressure remains constant.

Velocity: Velocity can effect on corrosion as the following:

- Corrosion of carbon steel in water in the near neutral pH range is governed by the rate of mass transfer of dissolved oxygen from the bulk fluid to the surface, if a porous surface hydroxide layer forms, the mass transfer rate might become limited by the diffusion through the porous film. This effect of velocity has ramifications for localized attack especially pitting and crevice corrosion, but the presence of fluid flow can some time be beneficial in preventing or decreasing localized attack.
- Fluid flow can cause a type of erosion of a surface through the mechanical flow of the fluid it self. This process called impingement the process in valves the removal of metal or alloy by the high wall shear stress created by the flowing fluid. When solid are present in the liquid, they can cause wear or solid erosion corrosion, the wear caused by relative movement of the solids with respect to the surface.

Concentration : Can effect on corrosion by adding small amounts of certain inhibitors or activators for example as a little of 0, 0023 mol/l of sodium nitrite (NaNO_2) or sodium sulfite (Na_2SO_3) can decrease the pit initiation rate of aluminum.

Similarly many organic inhibitors cause a drastic decrease in corrosion rate at very low concentration especially for iron in acidic solution with

no benefit observed up on increasing the inhibitors concentration.
All of these inhibitors tend to interact with the surface in the following:

- A guttering of a finite amount of impurity in the solution (hydrazine).
- By the oxidation of the surface (nitrite or chromate).
- Adsorption on the finite surface area to block corrosion (many organic inhibitors in acid). [10]

2.3.2 Soils Variables.

Several variables have been identified to have an influence on corrosion rates in soil as the following:

Water: In liquid state, represent essential electrolyte for electrochemical corrosion reaction. A distinction is made between the saturated and unsaturated water flow in soils, the unsaturated water flow represents movement of water from wet areas towards dry soil areas. The saturated water flow depends on the following:

- Pore size and distribution.
- Texture.
- Structure.
- Organic matter

pH: Soil in natural cause has a pH range from 5-8. In this range it's not having affecting corrosion rates. Where more acidic soils represents a serious risk to construction materials such as steel, cast iron and zinc coatings.

Soil acidity is produced by the following:

- Mineral loading
- Industrial wastes.
- Acid rain.
- Certain forms of micro-biological activity

Alkaline soils tend to have high contents of sodium, potassium, magnesium and calcium. Where the magnesium and calcium tend to form calcareous deposits on buried structures for protective against corrosion.

Chloride level: Chloride ions are generally harmful, as they participate directly in anodic dissolution reactions of metal and their presence tends to decrease the soil resistivity. The chloride ion concentration in the corrosive aqueous soil electrolyte will vary, as soil condition alternate between wet and dry.

Chloride may be found naturally in soils as results of the following:

- A brackish groundwater and historical geological sea beds.

- From external sources such as de-icing salts applied to roadways.

Resistivity: Is used as a brood indicator of soil corrosivity. Since the ionic current flow is associated with soil corrosion reactions, high soil resistivity will arguable slow down corrosion reactions. The resistivity of soil it generally decreases with increasing water content and concentration of ionic species. A high soil resistivity along will not guarantee absence of serious corrosion.

Degree of aeration: The oxygen concentration decreases with increasing depth of soil. In neutral or alkaline soils, the oxygen concentration has an important effect on corrosion rate due to its participation in the cathodic reaction.

In the presence of certain microbes corrosion rates can be very high, even under anaerobic conditions.

Excavation can obviously increase the degree of aeration in soil.

Sulfate level: Sulfates are generally considered to be more benign in their corrosive action towards metallic materials. However concrete may be attacked as a result of high sulfate levels. The presence of sulfates does pose a major risk for metallic materials in the since that sulfates can be converted to highly corrosive sulfides by anaerobic sulfate reducing bacteria.

Redox potential: The redox potential essentially is a measure of the degree of aeration in a soil. A high redox potential indicates a high oxygen level. Low redox values may provide an indication that conditions are conducive to anaerobic micro-biological activity.

Microbiologically influenced corrosion (MIC): Corrosion that influenced by the presence and activities of microorganisms and or their metabolites is refers to microbiologically influenced corrosion (MIC).

Rapid corrosion failures have been observed in soil due to microbial action And its becoming increasingly apparent that in most metallic alloys.

So that bacteria, fungi and other microorganisms can play a major part in soil corrosion. [11]

2.4 Causes of pipeline corrosion.

There are many causes can attribute corrosion in pipeline and they are as the following:

Dissimilar soils: Pipeline even it purled and has short length it will almost inevitably encounter soils that have varying compositions. When a dissimilar soils traverses in steel pipeline, the particular soil electrolyte will often assume galvanic potential that is some what different from the potential and portions of the same pipeline traversing dissimilar soils along the pipeline route. Such galvanic potential differences between different areas of the single pipeline can occur on (a macro scale) that is over many miles in the route of the pipeline or on (a micro scale) with in inches of each other or even over shorter distances.

Differential aeration: Is also referred as concentration cell corrosion. When the pipeline traversing soils that have varying levels of oxygen Concentration will be subjected to corrosion cell activity where the portion of steel pipeline of lowest oxygen concentration area is anodic to other area of pipeline where a greater concentration of oxygen.

Dissimilar metals: When the pipeline having dissimilar materials of construction wear they in contact with common electrolyte with no electrical isolation between the two metals, will subject to intense corrosion ,where the metals how have an highest in galvanic series will be anodic to the metals lowers in the series. An example of dissimilar metal corrosion is the use of magnesium alloys or Aluminum alloys in sea water and zinc as anodes (galvanic) for the protection of the structures of pipeline.

New and old steel pipe: New steel or old steel that has been mechanically restored to shiny surface, the condition will exhibit potential of a new steel will be between (-0.5 to -0.8) V. where the old or rusty steel will have potential between (-0.2 to -0.5) V. With bright, new steel pipe that is electrically connected to older or super-facially rusted steel pipe in an electrolyte, galvanic potential differences. As great as 0.5 V which can exist new pipes anodic to the older or rusty pipe and this will initiate corrosion in the pipe.

Bacteriological corrosion: Its microorganisms existing in the pipeline trench can affect either directly or indirectly on the control of corrosion. Bacteria that oxidize sulfuric can exist can exist aerated environments.

These bacteria (Thiobacillus Trioxides) consume oxygen and oxides sulfides in to sulfates such as sulfuric acid (H_2SO_4). By their metabolic process, these bacteria can create concentrations of the H_2SO_4 as high as 10%.

Another type of bacteria is anaerobic bacteria, which thrive in the absence of oxygen are sulfate reducing organisms that consume hydrogen and cause aloes of polarization at the steel pipeline surface. This loss of polarization can make the attainment of successful cathodic protection much more difficult.

Interference-current effects: It's referred to as stray – current effects. They usually occur when the direct currents associated with foreign metallic systems (one not direct associated with the pipeline of concern)

use of pipeline steel as preferential conductor in their source. When this occurs, the current will couple to the pipeline steel from the soil and flow longitudinally on the steel to allocations where they discharge from the steel to the adjacent soils in order to complete their circuit the hazard to the affected pipeline is primarily to the steel in the locations where current discharges to the earth, it is in essence creating anodic areas with attendant loss of pipeline.

There are two types of interference-current activity:

- Steady state.
- Transient state.

Steady state: in this type the magnitude of the interference current is essentially a constant, such currents are usually incurred by the operation of other nearby impressed. Current cathodic protection system.

Transient state: in this type the source of stray current is the operation of attraction system that uses direct current (dc) series motors for propulsion and the rail system as the return leg to their dc power source. [12]

2.5 Corrosion control and prevention methods for pipeline.

2.5.1 Internal and external pipelines coatings.

The coating provides a protective barrier against an extremely corrosive environment. However, coatings can be classified by their location on to protected pipeline to internal and external as the following:

Innerkote coating: Innerkote is a liquid paint coating using for internal gas flow and drinkable water pipeline. Coating is applied by spraying it onto the internal surface of steel pipelines for protection against corrosion and surface roughness reduction.

This coating is called Innersole and is commonly applied to gas pipelines for flow friction reduction, because the smooth finishing of surface will allows compression stations to save power.

Innersole can be used for different purposes by choosing the type of liquid paint and application method.

The procedures of the coating are as the following:

Internal surface of the pipe is first blast cleaned. Liquid paint is then sprayed with airless gun to form a uniform smooth layer. Paint is then cured either in oven or open air if an environment condition is permit. The typical coating thickness of Innersole is $65+100\mu\text{m}$ for gas transportation and $200+500\mu\text{m}$ for water or other liquids.

Tubekote coating: Tubekote is liquid or powder coating used for coating internal pipelines used for transport of oil, gas, water, industrial corrosive liquids. It is used for oil production pipelines, drilling and production tools such as drill pipes, sucker rods, packers, pumps, tubing installed in wells with corrosion problems, it is used in connection with special Joints for girth weld protection or for the Zap-Lock installation method.

The procedures of the coating are as the following:

Surface contamination removal blast cleaning to near white condition. Liquid epoxy is applied as epoxy primer by spray nozzle followed by hot curing. Where curing in oven, after that the epoxy primer application heating. Epoxy powder application by electrostatic spray guns is applied with thickness up to $200\mu\text{m}$.

Thickness is then being up to $400\mu\text{m}$ for both types, after that, they are cooled and subjected to 100% holiday free test.

Mortarkote coating: it is internal cement mortar pipe lining for steel and cast iron pipes and transporting drinkable water. This cement mortar lining is extends the service life of the pipeline by minimizing leaks due to internal

corrosion activity. Internal lining would not mitigate external corrosion activity, which would progress until the water pressure breaks the lining.

The procedures of the coating are as the following:

The internal surface is cleaned to remove any pipe residue of the mill manufacturing process, dirt and foreign particles. Then the cement mortar, properly prepared in a mixer. A mixer is then poured onto the internal surface of pipe by a boom equipped with a dispenser or a rotating spray head. In case of pouring the pipe is then rotated at high speed to distribute and homogenize the mortar and remove water excess. In the case of spraying the mortar surface is smoothened and leveled by a set of flexible towels gently rotating around the boom. Pipes are finally stockpiled for mortar setting and hardening.

Nylonkote coating: its internal pipe coating of Fusion Bonded Polyamide powder (Nylon 11) is suitable for potable water and oil transportation. Nylonkote is a thermoplastic material applied to the internal surface of steel pipelines for corrosion protection.

The procedures of the coating are in the following:

The internal pipe surface is first blast cleaned. The surface is then primed with an epoxy resin followed by heating and primer curing. When the pipe is hot, nylon powder is spray applied by electrostatic guns to melt and form a uniform and glossy layer. Nylonkote typical thickness is 200µm.

Wrapkote coating: its Asphalt or Coal Tar Enamel pipeline coating, applied to the external surface of pipes with inner and outer glass fiber reinforcement.

The coating thickness and number of inner wraps depend on the type Of service the pipe is intended for. The total coating thickness varies between 3, 5 mm and 7 mm.

The procedures of the coating are as the following:

Pipes previously preheated and cleaned. Pipes are first coated with bitumen or coal tar based primer and then allowed to dry. Then on the rotating and advancing pipe, bitumen /coal tar layer is applied while wrapped with one or two layers of glass cloth reinforcement. A layer of bitumen/coal tar impregnated glass cloth is then applied as a final and mechanical protection. The pipes after coating may be lime-coated to prevent sticking during storage and to minimize softening under sunlight.

Plastykote coating: It's anticorrosion pipe coating of extruded polyethylene/polypropylene jacket. Plasticity is a thermoplastic coating applied to steel pipeline as anticorrosion protection.

Plasticity mechanical resistance is a preference when there are risks of particularly severe coating damages. Different types are available according to pipelines service requirements are as the following:

- Two layer polyethylene.
It consists of an outer polyethylene layer and a bonding layer of copolymer adhesive which provides adhesion to steel surfaces. Two layer polyethylenes is suitable for service temperatures up to $60^{\circ}+80^{\circ}\text{C}$ depending on the type of materials used (LDPE –MDPE-HDPE). Typical coating thickness is from 1, 2 mm to 3 mm.
- Three layer polyethylene & polypropylene.
For polyethylene It's use to improve anticorrosion performance and adhesion. Additional layer of epoxy primer is sprayed onto pipe surfaces prior to the adhesive layer and polyethylene top layer application.
 - Three layer polyethylenes is suitable for service temperatures from 60 to 80°C (85°C peaks) depending on the types of materials used (LDPE –MDPE-HDPE).
Typical coating thickness is from 1, 2 mm to 3 mm.
If a wider service temperature range and high stiffness is required, adhesive and top layers, applied over primer layer, are based on polypropylene instead of polyethylene.
 - Three layer polypropylene is suitable for service temperatures up to 120°C (130°C peaks).
The application method is similar for all plasticity coatings. The pipe surface is blast cleaned, heated and then sprayed with epoxy primer.
(Except two layers), followed by the application of adhesive copolymer and polyolefin polymers, wrap or annular extruded, one over the other.

Powderkote coating: its anticorrosion pipeline coating of fusion bounded epoxy powder (FBE).

Powderkote is a powder epoxy thermosetting coating applied for anticorrosion protection to steel pipeline. The typical thickness of powderkote is $350+450\text{ }\mu\text{m}$. The powderkote it's suitable for pipeline service temperature up to 94°C .

The procedures of the coating are as the following:

The pipe is first blast cleaned and heated. The epoxy powder is sprayed by electrostatic guns. Then the epoxy will melt and form a uniform layer that hardness within a minute from application.

Purtarkote coating: It's Polyurethane Tar liquid paint for external anticorrosion pipe coating.

Purtarkote is double component polyurethane Eastover coating mixed with coal-tar, spray applied, for anticorrosion protection of steel pipelines.

The procedures of coating are as the following:

The pipe surface is first blast cleaned and preheated. Polyurethane tar is sprayed onto the pipe surface to required thickness. The separate components are independently pumped to the spray tip where a static mixer is installed for perfect blending. The standard thickness of purtarkote is 2 mm and it is appropriate for pipeline service temperatures up to 65°C. [13]

2.5.2 Control corrosion by material selection.

There are many ways we can use them to prevent or delay initiating of corrosion on pipeline and one of these ways is material selection.

We have to choose material for pipeline after us aspects the following factors:

- Safety
- Structural integrity.
- Operating life.
- Economic considerations.

After us aspect the last factor and taken in to account acted upon the following material:

Carbon steel: It's the almost exclusive choice of pipeline designers for using in transport of natural gas, crude oil and water over distances of hundreds of fetses or hundreds of miles. It is also the case of piping systems that are use to distribute natural gas, water, water refined liquids, and so on.

Cast iron: It's extensively used in water and natural gas distribution systems. In recent years the nonmetallic materials have found application in natural gas distribution systems as carrier vehicles and as liners for restoring failed metallic piping to service with out the need for trenching and replacement.

Carbon steels: Carbon steels and, in certain type's service or environmental conditions, alloy steels are by the far the most commonly used pipeline materials of construction.[12]

2.5.3 Protective coatings.

Coating is used to control corrosion by isolating the external surface of the underground or submerged piping from the environment, and excellent coatings have the following characteristics:

- Effective moisture barrier
- Good adhesion to the pipe surface.
- Effective electrical insulation.
- Ability to resist holidays with time.
- Ability to maintain substantially constant electrical resistivity over time.
- Ability to resist damage during handling, storing, age, and installation.
- Resistance to disbanding.
- Resistance to chemical degradation.
- Ease of repair.
- Retention of physical characteristics. [12]

Many types of organic coatings and lining using to protect pipeline against corrosion, and they can be classified in to five groups:

- Paint coatings
- Tape coatings
- Powder coatings
- Rubber coatings.
- Plastics coatings

For the paint coatings we have the following:

Liquid epoxies and phonemics: This system is used for larger-diameter pipe and operation temperatures in the 95°C range. Generally, epoxies have an amine or a polyamide curing agent and require a near-white blast cleaned surface. Coal tar epoxies have coal tar pitch added to the epoxy resin. Coal tar epoxy cured with a low molecular weight amine is especially resistant to an alkaline environment. Some coal tar epoxies become brittle when exposed to sunlight. [12]

- **High Build Epoxy Coating paint:** it is Brush applied high build epoxy coating is protective coating for pipelines in buried or immersed applications. Are a100% solids, two component epoxy coating system which specially use for exterior coating applied.
Typical uses of the brush FBE coating is:
 - It's used as a direct – to –metal corrosion and abrasion resistant coating.
 - It's used as a rehab coating on steel pipelines and at girth welds.
 - It's used as touch – up material for mainline coatings.

- It's use as excellent coating for pipeline valves, fittings, and bends.

Features of the brush FBE coating is:

- 100% solids.
- Environmentally safe.
- High build in a single coat.
- Excellent adhesion to grit blazed steel – an ideal mainline corrosion coating for pipeline.
- Superior adhesion to Fusion Bonded Epoxy coatings–ideal coating for joint protection and repair of FBE coating pipe.
- Sets and cures over a broad temperature range
- Excellent chemical and abrasion resistance.
- Easily applied with brush or roller.

Surface preparation before coating it could be by two methods:

- Direct – to – steel
This will be by remove all visible deposits of oil, grease and other contaminates by solvent washing in accordance with abrasive blast surfaces to near white or better with a 2- 4 mil blast profile.
- On cured pipe coating.
This will be by remove gloss on surface by light abrasive blasting or power tool. All surfaces to be coated must be dry, free of moisture, soil, dust and grit at the time the coating is applied also all weld must be removed from the surface.

The procedures of the coating:

Mix each of the based and hardener to an even consistency.
Add the hardener to base mix until an even color is an achieved, making sure all sides of container are scraped.
Immediately pour mixed material onto surface and brush, trowel or roll to required mil thickness.

A wet film thickness gauge shall be used to measure mil thickness.
If surface temperature falls below 50°F (10°C), surface must be preheated to maintain good application properties.
Preheat may be achieved with a propane torch or induction coil.
Base component shall be kept warm, at a minimum 68°F (20°C), to achieve easy mixing properties. [14]

Table1: Typical properties of high build Epoxy coating brush applied [14]

Properties	Data
Solids Content	100%
Mixed Material - mixed at 77°F (25°C)	
Specific Gravity	1.46
Viscosity	Thixotropic liquid
Color	Cream yellow
Handling Time	2 hours
Mixing Ratio by Volume	3 parts base, 1 part hardener
Adhesion to Steel	2300 psi
Adhesion to FBE	1100 psi
Impact Resistance	Excellent
Hardness (ASTM 2240)	Shore D min 75
Service Temperature	-40°F to 150°F (-40°C to 65°C)
Glass Transition	150°F (65°C)

- **Pragokor Metal E (zincethylsilicate paint):** Zincethylsilicate paint it consists of two component, modified and solvent based zincethylsilicate paint, with high content of metallic zinc, for anticorrosion protection of steel structures in aggressive environments, being cured by solvent evaporation and atmospheric moisture.

Typical uses: It's use as a single coat or top coated primer for long-term steel structures protection in aggressive atmospheres or other environments of the pH value within 5,5-10,5 for neutral salts or sea water.

It's use for the structures exposed to high humidity or water spraying for mining or under earth structures.

Its can be used also for building structures (bridges), technological structure for columns, exchangers, pipeline bridges, pipelines, furnaces, mills, crushers.

It can be used as protective tank linings for storage of crude oil and it refined products oils and fuels. And be used as a local protection of the burnt metallize galvanized areas on the structures after welding.

Properties: The zincethylsilicate paints have the following properties: It is inorganic coating with temperature resistance up to 400°C as a single coat and that be even enhanced by an aluminum pigmented silicone topcoat. Excellent temperature shocking durability.

Long term corrosion protection at least (15 years) in very aggressive atmospheres without any maintenance. The life can be raised to more than 15 years by top coating with organic paints such as epoxy, coal tar epoxy polyurethane, vinyl. Very high hardness and abrasion resistance after complete curing by atmospheric humidity. Can be even applied at temperatures around 0°C. Entire resistance against ultra violet radiation (UV) sun and biodegradation.

Excellent protection functions, in an electrochemical way, against under rusting at tiny imperfections or mechanical damage of the coating. [15]

Table 2: Typical properties of the Pragokor Metal E [15]

Finish	metallic grey, dull
Theoretical coverage at 60 µm DFT (calculated)	4,1 m ² /kg (11 m ² /litre)
Solids content	79,7 % by mass
Recommended overall dry film thickness (DFT)	60-80 µm-when applied by spraying 1 Coat and by brush 2 coats of thinned paint
Pot life	8 hours (at 20°C)
Mixing ratio liquid-powder components by weight	1 : 3
Density	2,7 kg/liter
Application	Airless or conventional air-assisted spraying, brush

For the tape coatings we have the following:

- **Mill – applied tape systems:** It's called as cold applied tape for normal construction condition and it's applied as three layer system:

- Primer and its function are to provide abounded medium between the pipe surface and the adhesive or sealant on the inner layer.
 - Corrosion preventive tape (inner layer), consists of plastic backing and an adhesive so this layer is the corrosion protective coating, therefore it must provide a high electrical resistivity, low moisture absorption and permeability, and an effected bond to the primed steel surface.
 - Mechanically protective tape (outer layer), the outer layer tape consists of a plastic film and an adhesive composed of the same tape or materials that are compatible with the inner layer tape, and the purpose of the outer layer is to provide mechanical protection to the inner layer tape and to be resistant to the elements during outdoor storage.[12]
- **Waxes:** Wax coatings have been in use for more than 48 years and still employed on a limited basis. Its use with a protective over wrap. The wax serves to waterproof the pipe, and the wrapper protects the wax coating from contact with the soil and affords some mechanical protection. [12]
 - **Polyurethane thermal insulation:** Its efficient pipeline insulation and its important as means of operating hot and cold service pipelines. Polyurethane insulation is generally used in conjunction with a corrosion coating, but if the proper moisture vapor barrier is used over the polyurethane foam, effective corrosion protection is attained.[12]
 - **Primer less Protective Tape Pipeline Coating:** Wrapped tape its protective coating tape consists of polyolefin (polyethylene and fusion bonded epoxy) used for protect and repair pipeline, fittings, bends, elbows and irregular configurations. It's coating tape with protective heat sensitive anticorrosion adhesive which effectively bonds to steel substrates and pipeline. With application of heat this coating tape shrinks down to fully encapsulate the protected substrate. [12]

Features: Easy installation, because the flexibility of the tape which allows it to protect both conventional substrate such as pipe as will as irregular transitions such as elbows and risers. Although the application of heat that specially formulated adhesive flows into all surface irregularities, forming a protective barrier against corrosion. Saving time and money, it's easy and quick to install without need to priming or special operator equipments. Because it's quick to install the labor costs are kept to minimum. Protection for long time, it's manufactured from material that provides high electrical resistivity, low water absorption and low moisture permeability.

This wrapped tape is much tougher than conventional tapes, so more effectively resisting abrasion and damage and that will extend lifetime. This tape can also be double or triple wrapped for extra mechanical protection when required. [16]

Table 3: Typical properties for primer less cross linked protective tape. [16]

Operating Characteristics	Standard	Units	Wrapped Tape
Pipeline operating temperature		°C °F	-40 to 53 -40 to 127
Resistance to soil stress			good
Main line coating compatibility			PE, FBE, PU
Softening point	ASTM E28	°C °F	72 162
Tensile strength	ASTM D638	MPA psi	20 2900
Elongation	ASTM D638	%	600
Hardness	ASTM D2240	Shore D	46
Abrasion resistance	ASTM D1040	mg	45
Impact	DIN 30 672	pass/fai I	pass
Water absorption	ASTM D570	%	0.05

For the powder coatings we have the following:

- **Fusion bounded epoxy coatings:** its heat – activated, chemically cured coating systems, and it's furnished in powdered form (Fusion bounded epoxy coatings). These coating is applied to preheated pipe surfaces at 218 to 244°C.

The epoxy coatings exhibit good mechanical and physical properties and are the most resistant to hydrocarbons, acids, and alkalis. The coating is applied to minimum thickness of 0.33 mm, in some applications, coating thicknesses range to 0.64 mm, with the restriction not to bend pipe coated with film thickness greater than 0.4 mm.

The advantages of the fusion bounded pipe coatings:

- They can not hide apparent steel defects; therefore, the steel surface can be inspected after it is coated.
- The number of holidays that occur is function of the surface condition and the thickness of the coating specified, were increasing the thickness minimizes this problem.
- Excellent resistance to the electrically induced disbandment of these coatings.[12]

In generally (FBE) it's Powder coating which have played a part in the protection of pipelines for almost 40 years and are being further enhanced to ensure that they will continue to play an important role in pipeline protection long into the future, the application of a thin film of the FBE is (350-600 micron) provided an excellent anticorrosion coating for the alkali conditions obtained from cathodically protected pipelines. The limitations of the system include relatively low mechanical resistance which can cause an increase in handling and laying costs for the pipeline during installation. More recently FBE-based coatings have undergone developments to improve mechanical resistance through the use of additional layers of modified FBE, liquid polyurethane or thermoplastic outer coats. In the 1960s, the Europeans adopted technology based on isolating the pipe from its environment through the use of a polyethylene PE coating. Over the years, this has developed from the initial sinter coat systems, which showed poor adhesion, through soft adhesive and hard adhesive two-layer systems.

In the 1980s liquid epoxy-primed three-layer systems were developed which led to the real breakthrough with the introduction of powder epoxy primers. These gave economic, environmental and performance advantages and converted most of the market to what is known as three layer PE coatings consisting of thin (60-120 micron) fusion-bonded epoxy, adhesive and polyethylene top coat. The thickness of the epoxy has increased in appreciation that better performance can be obtained with increasing the coating layer more than 200 microns of epoxy. This increase in the epoxy thickness view to enhance mechanical and anti-corrosion properties of the protection system through multi-layered complex coatings involving a variety of materials and application methods. But as the search for oil and gas takes the industry to more remote areas, deeper water, deeper wells and contaminated oils, the demands for protection of the transport system are increasing all the time. An ever more competitive world drives the technologies for new solutions to reduce total lifetime costs but without loss of confidence. A potential example of this is the use of powder/powder or powder/liquid systems. They improve the mechanical properties of FBE which may yield a lower material cost than three-layer polyethylene and/or reduce

the layer cost compared to standard FBE while having a proven track record of FBE as an anticorrosion coating.

The FBE powder products for use in the pipeline include:

- High abrasion-resistant coatings for thrust bore protection.
- Improved surface tolerance.
- High glass transition temperature epoxy (150 -160 deg C°) with flexibility to facilitate reel lay operations.
- Energy absorbing coatings for impact and backfill protection. [17]

For best results of Fusion Bonded Epoxy coatings we must control following critical steps:

- Preparation of pipe prior to abrasive cleaning.
- Abrasive cleaning.
- Final cleaning and final inspection.
- Surface conditioning.
- Preheating of the pipe
- FBE Coating Application.
- Post treating.
- Final Inspection and Quality Control
- Coating Repair procedures.
- Pipe Handling. [18]

Fusion Bonded Epoxy: Fusion bonded epoxy can be classified to three groups:

- Fusion bounded epoxy (FBE) single layer.
- Dual layer (FBE).
- Three layer FBE/polyolefin adhesive/polyolefin.

Fusion bounded epoxy (FBE) single layer: This type of coating for external pipeline for oil and gas pipeline systems. Is used since 1960's for pipeline coating. FBE coating is a thermosetting resin applied in the form of a dry powder onto the heated surface of steel pipe.

The cured film is homogeneous, with optimum adhesion to the steel and Good resistance to chemical reaction. The correct surface temperature allows the fusion of the powder and assures the polymerization process, which is completed with a final cooling stage, so the epoxy powder achieves an extremely hard surface with excellent adhesion to the steel surface.

The coating application system consists of numerous electrostatic

spray guns that uniformly deposit the epoxy powder onto the blast cleaned chemically treated and preheated surface of the pipe. [19]

Dual layer (FBE): Unique Fusion Bonded Epoxy System is applied as a powder base coat and powder top coat simultaneously with the typical application system for the conventional FBE.

It has excellent impact resistance and abrasion properties which combined with its good flexibility provide good protection against possible damage to the coating during pipe transportation and pipeline construction.

The steel pipe is coated with a dual powder system as following:
A regular Fusion Bonded Epoxy resin layer provides corrosion protection. Then a second layer of a modified FBE resin is applied for a rough finishing. The main purpose of this type of coating system is to provide a rough, non-slip surface for a pipeline that will subsequently be concrete coated.

Three layer FBE/polyolefin adhesive/polyolefin: It's consisting of three layers; FBE, polyolefin adhesive, polyolefin.

Polyolefin coat can be either polyethylene or polypropylene, where:
Three layer Polyethylene, suitable for temperatures between 40° C and 80°C.

Three layer Polypropylene, suitable for temperatures up to 120°C.
The polyethylene 3 layer system provides physical and mechanical advantages, whilst the polypropylene system also ensures performance at high temperatures.

Polyolefin adhesive (rough coat) or polyolefin (rough finish) both of them consisting of polyolefin powder which applied during shop application.
A rough coat is used to:

- To improve friction between the FBE and a cement weight coating.
- To improve traction for lay barge operations and improve safety.

Product procedure:

Pipe is blast cleaned. Then it preheated and one layer of Fusion Bonded Epoxy powder (FBE) is then applied. While the FBE is still in its gelled state a copolymer adhesive layer is extruded onto the FBE. Then it followed by a layer of extruded polyethylene or polypropylene until the desired thickness is obtained.

Advantages of Three Layer Coatings:

- Excellent corrosion resistance.
- Strong steel adhesion: 20 times more adhesive than traditional plastic tape systems.

- Excellent cathodic disbandment test results.
- Optimum impact resistance.
- Free of holidays: The hot extrusion method used to perform the coating assures a continuous and a uniform thickness profile, without air bubbles and holiday free.
- High dielectric resistance.
- Superior bends ability for laying
- Impermeability: Both the high density Polyethylene and the Polypropylene have low water permeation characteristics which are less than those of any other pipeline coating system. [18]

Table 4: Typical properties for extruded polyolefin coatings [6]

Property	Typical Value Polyolefin Resin	Butyl Adhesive
Density	Minimum 0.95 g/cm ³	Minimum 1.00 g/cm ³
Flow Rate	Maximum 0.75 g/10 minutes	Maximum 8.00 g/10 minutes
Tensile Elongation	500%	
Tensile Strength	Minimum 19 MPa (2,800 psi)	
Hardness	60 (Shore D)	
Water Absorption	Maximum of 0.02% for the total system	

For the rubber coatings we have the following:

- **Bituminous enamels:** This type is formulated from coal tar pitches or petroleum asphalts. Coal tar and asphalt enamels are available in summer and winter grades. Enamel systems can be designed for installation and use with in an operating temperature range of 1 to 82°C. When temperatures fall below 4.4° C added precautions should be taken to prevent cracking and disbanding of the coating during field installation. Enamels are affected by ultra violet rays, hydrocarbons. Bituminous enamel coatings are available for all sizes of pipe.

The use of enamels has declined for the following reasons:

- Reduced number of suppliers.
- Restrictive environmental and health standards from the occupational safety and health administration.

- Increased acceptance of plastic coating materials.
 - Alternative use of coating raw materials as fuels.[12]
- **Asphalt mastic pipe coating:** Its mixture of sand, cursed lime stone, and fiber bound to gather with select air-blown asphalt. The coating hick is (12.7 to 16 mm, it's the thickest of the corrosion coating. This type is cost effective for offshore installations. It use for temperature operation within range of 4.4 to 88°C. It's not recommended to use above ground or in hydrocarbon-contaminated soils. This type is used for out side diameter pipe range 11.4 – 122 cm. [12]
- **Coal tar enamel coat:** Coal tar enamel pipe coating systems it's for external purpose. Coal tar enamels have been used as a pipeline coating since the1930's, its have some advantages like good resistance to moisture absorption, are easy to apply to the girth weld zone, and a good coefficient of friction, but it have also disadvantage like subject to oxidation and cracking, soil stress has been an issue, limitations at low application temperatures, environmental and exposure concerns, associated with corrosion and stress crack corrosion failures. [6]

Table 5 : Typical properties for coal tar enamel coatings [6]

Property	Typical Value
Thermal Conductivity	0.16 W/m-K (1.1 BTU/ft2/h/oF/inch)
Electrical Resistance	1 x 10 ¹⁴ ohm-cm
Dielectric Strength	>10 V/μm (250 V/mil)
Water Absorption	2% or 0.3 g/30 cm ² (0.1 oz/50 in ²)
Coefficient of Friction	0.59 to 0.91

For plastic coating we have the following:

- **Extruded plastic coatings:** This type is based on the methods of extrusion, with the additional variations resulting from the selection of adhesive.

There are two methods of extrusion:

- The crosshead or circular die.
- The side extrusion or T-shaped die.

The four types of adhesives are asphalt –rubber blend, polyethylene copolymer, butyl rubber adhesive, and polyolefin rubber blend. [12]

2.5.4 Cathodic protection for pipelines.

Cathodic protection effectively protects underground or submerged metallic structures (iron or steel) such as underground pipelines, storage tanks, and the interior of water storage tanks, ocean pilings. Through the use of a negative potential applied by an external source to the structure. Commonly, once the structure has been made sufficiently negative, environmental corrosion (soil or moisture) is resisted.

Principle of cathodic protection: The basic principle of CP is simple. A metal dissolution is reduced through the application of a cathodic current. Cathodic protection is often applied to coated structures, with the coating providing the primary form of corrosion protection. The CP current requirements tend to be excessive for uncoated systems. Cathodic protection, in conjunction with protective coatings, is the primary method of defense against corrosion on major cross country pipelines. Cathodic protection requires the constant delivery of a steady electrical charge. In a cathodic protection system, the pipeline is negatively charged, acting as a cathode. A positively charged sacrificial anode completes the circuit. In this system, oxidation occurs in the anode while the cathode—that is, the pipeline—is protected from corrosion. [20]

Cathodic protection surveys on sub sea pipelines

Over the last 18 years, cathodic protection have surveyed over 20,000 kilometers of sub sea pipeline for some of the best international oil industry. Their worldwide experience covers South Atlantic, Gulf of Mexico and Sea of Japan. Accurate information about the condition of cathodic protection along the length of a pipeline is vital to the operator who needs to make informed decisions about maintenance or repair.

Function of critic: Through the ability to gather data and interpret it, in the light of their comprehensive experience, can provide the answers to questions concerning the performance of the system, the severity of dielectric coating breakdown and the remaining life of the anodes. To obtain this data, it's unique in offering the option of Trailing Wire (TW), Remotely Operated Vehicle (ROV) or hybrid ROV TW survey techniques, according to the nature of the pipeline to be inspected.

The procedure of the (ROV): The system is based around two networked PCs which record the survey data as well as displaying it graphically in real time on a color screen. The computers can also output comprehensively annotated hardcopy for analysis. Trouble spots can be identified whilst the survey is in progress so that additional information can be gathered in preparation for repair or maintenance.

After completion of survey sections, provisional plots and a status summary are available within hours.

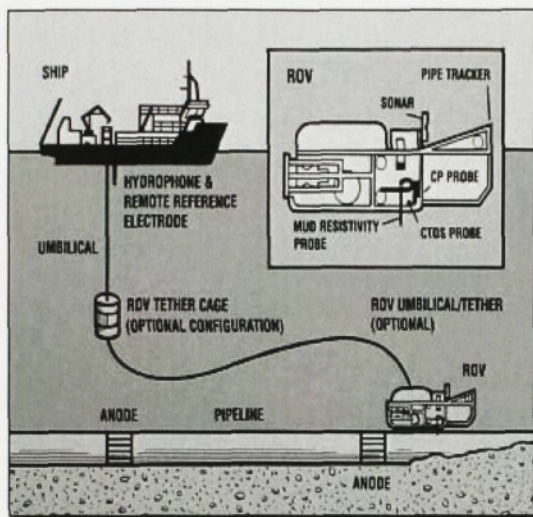


Fig 1: Remotely operated vehicle survey

Advantages of the ROV System:

- Highly detailed results.
- Accurate to within ± 2 mV.
- Locates and measures the magnitude and direction of the current flow.
- Calculation of anode output and life predictions.
- Compiled with video/bathymetric survey data which provides a full picture of pipeline status

The function of the trailing wire system: The 'trailing wire' system is a fast and convenient method of undertaking cathodic protection surveys on buried pipelines where ROV methods are not practical or cost effective. Pipeline potential is measured using a 'towed fish' which is maintained as close to the pipeline as possible, and a 'trailing wire' connected to the pipeline at a riser.

In common with the ROV method, the system is based around two networked PC compatible computers. The system measures variations in potential and overall levels of protection, thereby providing an overall illustration of trends along the pipeline.

The 'trailing wire' technique is ideally suited to in-field pipelines up to 40km in length. Beyond this length, operational difficulties and the limitations of the system can make the method ineffective. [21]

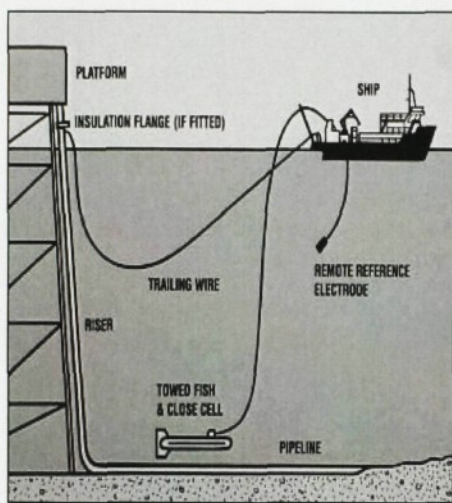


Fig 2: Trailing wire survey systems

2.5.5 Corrosion Inhibitors.

A corrosion inhibitor may be defined, in general terms, as a substance which when added in a small concentration to an environment effectively reduces the corrosion rate of a metal exposed to that environment. The use of chemical inhibitors to decrease the rate of corrosion processes is quite varied. However they often play an important role. In the oil extraction and processing industries, for example, inhibitors have always been considered to be the first line of defense against corrosion.

Inhibitors are chemicals that react with a metallic surface, or the environment this surface is exposed to, giving the surface a certain level of protection. Inhibitors often work by adsorbing themselves on the metallic surface, protecting the metallic surface by forming a film. Inhibitors are normally distributed from a solution or dispersion.

Inhibitors slow corrosion processes by either

- Increasing the anodic or cathodic polarization behavior.
- Reducing the movement or diffusion of ions to the metallic surface.
- Increasing the electrical resistance of the metallic surface.

Passivity inhibitors: (activators) cause a large anodic shift of the corrosion potential, forcing the metallic surface into the passivity range. There are two types of passivity inhibitors:

- Oxidizing anions, such as chromate, nitrite and nitrate, that can passivity steel in the absence of oxygen.
- Non oxidizing ions such as phosphate, tungstate and molybdate that require the presence of oxygen to passivity steel.

These inhibitors are the most effective and consequently the most widely used. Chromate based inhibitors are the least expensive inhibitors and were used until recently in a variety of applications, e.g. recirculation-cooling systems of internal combustion engines, rectifiers, refrigeration units, and cooling towers. Sodium chromate, typically in concentrations of 0.04-0.1% was used for these applications.

Evaluation of Corrosion Inhibitors: In choosing between possible inhibitors, the simplest tests should be done first to screen out unsuitable candidates. The philosophy of initial screening tests should be that poorly performing candidates are not carried forward. An inhibitor that does poorly in early screening tests might actually do well in the actual system, but the user seldom has the resources to test all possible inhibitors. The inhibitor user must employ test procedures that rigorously exclude inferior inhibitors even though some good inhibitors are excluded.

Effects of Inhibitors on Corrosion Processes: In acid solutions the anodic process of corrosion is the passage of metal ions from the oxide-free metal surface into the solution, and the principal cathodic process is the discharge of hydrogen ions to produce hydrogen gas. In air saturated acid solutions, cathodic reduction of dissolved oxygen also occurs, but for iron the rate does not become significant compared to the rate of hydrogen ion discharge until the pH exceeds a value of 3. An inhibitor may decrease the rate of the anodic process, the cathodic process or both processes.

The change in the corrosion potential on addition of the inhibitor is often a useful indication of which process is retarded. Displacement of the corrosion potential in the positive direction indicates mainly retardation of the anodic process (anodic control), whereas displacement in the negative direction indicates mainly retardation of the cathodic process (cathodic control). Little change in the corrosion potential suggests that both anodic and cathodic processes are retarded.

The usage of anodic and cathodic inhibitors for acid cleaning of Industrial equipment. The combined action of film growth and deposition from solution results in fouling that has to be removed to restore the efficiency of heat exchangers, boilers and steam generators.

Pourbaix or E-pH diagrams: Indicate that the fouling of iron-based boiler tubes, by (Fe_3O_4) and (Fe_2O_3), can be dissolved in either the acidic or alkaline corrosion regions. In practice, inhibited hydrochloric acid has been repeatedly proven to be the most efficient method to remove fouling. Four equations are basically needed to explain the chemistry involved in fouling removal.

The following three of those equations represent cathodic processes:

- $\text{Fe}_2\text{O}_3 + 4 \text{Cl}^- + 6 \text{H}^+ + 2 \text{e}^- \rightarrow 2 \text{FeCl}_{2(\text{aq})} + 3 \text{H}_2\text{O}$
- $\text{Fe}_3\text{O}_4 + 6 \text{Cl}^- + 8 \text{H}^+ + 2 \text{e}^- \rightarrow 3 \text{FeCl}_{2(\text{aq})} + 4 \text{H}_2\text{O}$
- $2 \text{H}^+ + 2 \text{e}^- \rightarrow \text{H}_2$

And one anodic process, i.e. the dissolution of tubular material

- $\text{Fe} + 2 \text{Cl}^- \rightarrow \text{FeCl}_{2(\text{aq})} + 2 \text{e}^-$

These equations indicate that the base iron functions as a reducer to accelerate the dissolution of iron oxides.

Since it is difficult to determine the end point for the dissolution of fouling oxides, an inhibitor is generally added for safety purpose. Both anodic and cathodic inhibitor could be added to retard the corrosion of the bare metal after dissolution of the fouling oxides. [22]

Corrosion: Pourbaix diagram: This diagram indicates if the situation is dangerous [22]

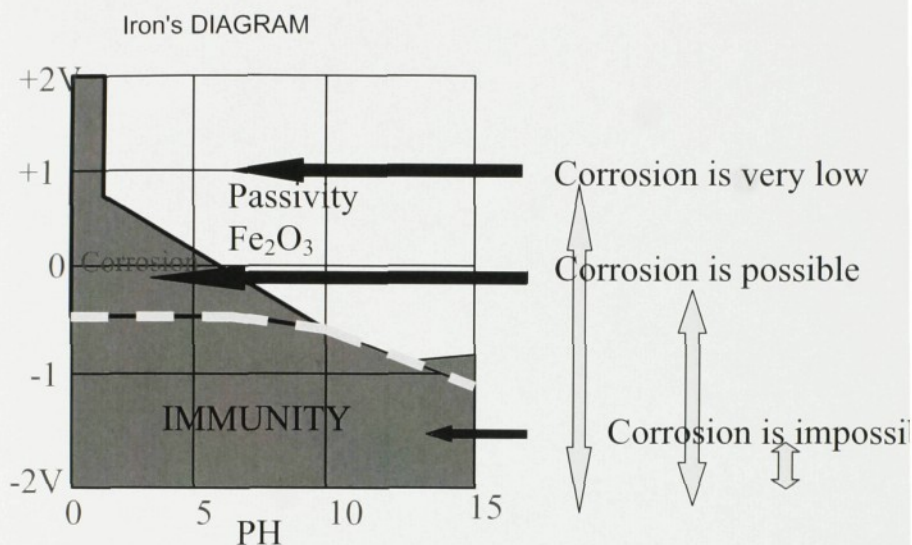


Fig 3: Pourbaix diagram

2.6 Methods of testing and monitoring of corrosion.

2.6.1 Planning and preparation of corrosion tests.

There are many steps before the corrosion tests and they are as the following:

- Test objectives.

The first and most important part of test planning is to define clear objectives because it determines the kind of information that would be the pertinent to the goals of the test. Typical corrosion test objectives include:

- Determining the best material.
- Predicting the probable service life of a product or structure.
- Evaluating the new commercial alloys and processes.
- Assisting with development of materials with improved resistance to corrosion.
- Conducting lot – release and acceptance tests to determine whether material meets specifications (quality control)
- Evaluating environmental variations and controls.
- Determining the most economical means of reducing corrosion.
- Studying corrosion mechanisms.

- Procurement of test materials

The procurement of test materials depends on:

- Metal composition and metallurgical condition, where mill fabricated material is best procedures for commercial alloy, but for the nonstandard alloys or metallurgical treatment are to be evaluated it may be economically impractical to have them fabricated with large production equipment.
- Metal form .metals is available in two basic forms: cast and wrought (worked) .these two forms should not be interchanged in testing.

- Preparation of test specimens.

The uniformity of the metal sample should be checked in advance as part of the plan for preparation of the test specimens.

- Selection of corrosive media.

- The corrosive behavior of a given material depends on the environment and the conditions of exposure as well as the condition of the test material.

- A laboratory tests can be misleading if appropriate
 - Corrosive media relating back to the test objectives are not used.
 - The corrosion investigator must do every thing possible to make the test reproducible by exercising explicit control over such environmental factors
- Assessment of corrosion damage.
The methods of assessment are limited only by the ingenuity of the investigator, who must try to find the best way to convey the practical meaning of the test results.
 - Reliability of corrosion test results.
Reliability of corrosion test results depends on:
 - Corrosion rate.
 - Specimen replication.
 - Precision and bias.
 - The unexpected. [23]

2.6.2 Selecting corrosion-monitoring methods. .

The most widely used method of corrosion monitoring involves the exposure and evaluation of the corrosion in actual test coupons (specimens).

In the selection of corrosion – monitoring method, a variety of factors should be considered:

- The purpose of the test should be understood by everyone concerned with the corrosion monitoring program.
 - The cost and applicability of the methods under consideration should be known.
 - Considering the reliability of the method selected.
 - The response time required to obtain the desired information from the method.
 - One of safety.
- Ultrasonic thickness measurements
Ultrasonic thickness can be used to monitor corrosion rates in situ.
 - Polarization resistance measurement. It uses to provide information in remaining thickness this technique provides also an estimate of the corrosion rate.
 - Measurement of corrosion potentials.
This measurement it use where an alloy could show both active and passive corrosion behavior in a given process stream. [24]

2.6.3 Laboratory corrosion tests.

There are many types of corrosion testing and they are as the following:

- Electrochemical methods of corrosion testing.
- Immersion tests.
- Corrosion cabinet testing.
- High temperature corrosion testing

Electrochemical methods of corrosion testing: While all laboratory corrosion tests require accelerating corrosion processes, only electrochemical tests can directly amplify the impact of corrosion processes. The main reason why this is possible is that all electrochemical tests use some fundamental model of the electrode kinetics associated with corrosion processes to quantify corrosion rates. The amplification of the electrical signals generated during these tests has permitted very precise and sensitive measurements to be carried out.

- Electrochemical methods for the study of localized corrosion.
 - Tests for evaluating the susceptibility of material to pitting and crevice corrosion include potentiodynamic tests, Galvanostatic tests, potentiostatic tests, scratch potentiostatic tests, triboelectrochemical methods, pit- propagation and electrochemical noise measurement.
 - Potentiostatic and Galvanostatic methods for localized corrosion. Potentiostatic methods can be used to overcome the inherent problems involving scan rate.
 - There are two general methods:
The first is mainly associated with the study of initiation.
The second is mainly associated with the study of propagation and reactivation.
 - The scratch-reactivation methods for localized corrosion. In this method, the alloy surface is scratched at a constant potential, and the current is measured as a function of time.
- Environmental cracking.

There are numerous electrochemical techniques in this area:

 - One of the techniques is for alloys that undergo passive film rupture and anodic dissolution.
 - The other is for alloys that are susceptible to hydrogen embrittlement.
- Stress corrosion cracking (SCC).

In this area of electrochemical technique, the scratch-

Reactivation technique has been used to distinguish between the case where an alloy is susceptible to localized corrosion, such as pitting, in general, once the passive film is mechanically disturbed at some potential, an electrochemical current can be measured that will decay back to a low level when reactivation has occurred. The electrochemical test is conducted in conjunction with the optical ellipsometric method to measure the rate of film growth. This combination offers a method of separating the two currents from one another because it is means of studying the kinetics of the film growth associated with reactivation.

○ Hydrogen embrittlement

It's used to study the parameters associated with the hydrogen embrittlement tendencies of metals and alloys, a then parallel faced wafer of the metal of interest is positioned between two separate polarization cells such that there is no electrolyte path between the two cells.

Adsorbed hydrogen is electrochemically generated through cathodic polarization and hydrogen evolution on one surface of the metal. The hydrogen that diffuses through the thin piece of metal is oxidized on the opposite surface by applying anodic polarization.

○ Electrochemical evaluation of protective coatings and films by the following:

- Numerous ac and dc electrochemical methods have been used to study the performance and the quality of protective coatings including passive films on metallic substrate and to evaluate the effectiveness of various surface pretreatments.

- Anodized aluminum corrosion test.

This test involves the cathodic polarization of the anodized aluminum surface by using a small cylindrical glass clamp on cell and a special (NaCl) solution containing cupric chloride (CuCl_2) acidified with acetic acid.

A large voltage is applied across the cell by using a platinum auxiliary electrode. The alkaline conditions created by the cathodic polarization promote dissolution at small defects in the anodized aluminum.

The coating resistance is decreased, more current begins to flow, and the voltage decreases. The cell voltage monitored for 3 min and the parameter cell voltage is multiplied by time is recorded. A similar test is a cathodic break down test. The test was designed for anodized aluminum alloys because the alkali created at large applied currents will promote the formation of corroded spots at defects in the anodized film.

- The electrolytic corrosion test.
It was designed for electrodeposits of principally nickel and chromium on less noble metal, such as zinc and steel. Metal is polarized to +0.3 V versus the standard calomel electrode. The metal is taken through cycles of 1 min anodic ally polarized and 2 min unpolarized. An indicator solution is then used to detect the presence of pits that penetrate to the substrate.
- The paint adhesion on ascribed surface (PASS) test
In this test the cathodic polarization of a small portion of painted metal .the area exposed contains a scribed line that exposes a line of underlying bare metals.
The sample is cathodically polarized for 15 min in 5% NaCl. At the end of this period, the amount of delaminated coating is determined from an adhesive tape pulling procedure.
- Electrochemical impedance spectroscopy
It's known also as electrochemical impedance technique and it offers an advanced method of evaluating the performance of metallic coatings and organic barrier coatings. The technique is quite sensitive to changes in the resistive capacitive nature of coatings. Also it's possible to monitor the corrosion rate by this technique.
- Limitations of electrochemical tests.
Electrochemical techniques are not always successful, so there are three specific keys to successful application of these techniques they:
 - The implementation of relevant environment.
 - The relevant alloy and surface preparation.
 - The relevant mechanical perturbations.
 - Without these essential features any electrochemical test designed to simulate. For example, a chemical plant process stream will be destined for failure. [25]

Immersion testing: The environmental conditions that must be simulated and the degree of acceleration that is required often determine the choice of a laboratory test. In immersion testing, acceleration is achieved principally by:

- Intensifying the conditions in order to increase corrosion rates, i.e. increasing solution acidity, salt concentration, temperature or pressure.

- Lengthening the exposure to the critical conditions that are suspected to cause corrosion damage. For example, if a vessel is to be batch-processed with a chemical for 24 h, then laboratory corrosion exposure of 240 h should be considered.
- Once the environmental conditions have been determined, and the test designed, then it should be repeated a sufficient number of times to determine whether it meets the desired standard for reproducibility.

Immersion tests can be divided into two categories:

- Alternate immersion tests: another variation of the immersion test is the cyclic test procedure where a test specimen is immersed for a period of time in a test environment, then removed and dried before being re-immersed to continue the cycle. Normally hundreds of these cycles are completed during the course of a test program.
- Simple immersion tests: basically small sections of the candidate material are exposed to the test medium and the loss of weight of the material is measured for a period of time. Immersion testing remains the best method of screening and eliminating from further consideration those materials that should not be considered for specific applications. But while these tests are the quickest and most economical means for providing a preliminary selection of best suited materials, there is no simple way to extrapolate the results obtained from these simple tests to the prediction of system lifetime. [26]

Corrosion Cabinet Testing: Cabinet testing refers to tests conducted in closed cabinets where the conditions of exposure are controlled and mostly designed to accelerate specific corrosion situations while trying to emulate as closely as possible the corrosion mechanisms at play.

Cabinet tests are generally used to determine the corrosion performance of materials intended for use in natural atmospheres. In order to correlate test results with service performance, it is necessary to establish acceleration factors and to verify that the corrosion mechanisms are indeed following the same paths.

Modern surface analysis techniques can be quite useful to ascertain that the corrosion products have the same morphologies and crystallographic structures that are typically found on equipment used in service.

There are basically three types of cabinet tests:

- Controlled humidity test:

There are different variations of creating and controlling fog and humidity in cabinets for corrosion testing of a broad spectrum of products, from decorative electrodeposited coatings to the evaluation of the corrosivity of solder fluxes for copper tubing systems. The basic humidity test is most commonly used to evaluate the corrosivity of materials or the effects of residual contaminants. Cyclic humidity tests are conducted to simulate exposure to high humidity and heat typical of tropical environments. The cabinet performing such tests should be equipped with a solid-state humidity sensor reading the current humidity condition and a feedback controller. The mechanism used to control the humidity moves chamber air via a blower motor and passes it over a heater coil in the bottom of the chamber with an atomizer nozzle fogging into this air stream.

- Corrosive gas test:

In these tests, controlled amounts of corrosive gases are added to humidity to replicate more severe environments. Some of these tests are designed to reveal and amplify certain characteristics of a material. There are many types of corrosive gas tests and they are:
The ASTM B775 (Test Method for Porosity in Gold Coatings on Metal substrates by Nitric Acid Vapor)

- B 799 (Test Method for Porosity in Gold or Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor) employs very high concentrations of corrosive gases to amplify the presence of pores in gold or palladium coatings.
- The moist sulfur dioxide test intended to produce corrosion in a form resembling that in industrial environments.
- A flowing of mixed gas test, that consists in introducing parts per billion levels of pollutants such as chlorine, hydrogen sulfide and nitrogen dioxide in a chamber at a controlled temperature. [27]

- Salt spray testing:

The oldest and most widely used cabinet test is Method of Salt Spray, a test that introduces a spray in a closed chamber where some specimens are exposed at specific locations and angles. Its accelerated tests for determining the corrodibility of nonferrous and ferrous metals as well as the degree of protection offered by butting inorganic and organic coatings on a metallic base.

The primary objective is to provide an easily performed acceptable standard for comparing the performance of materials and coatings. The concentration of the NaCl solution has ranged from 3.5 to 20%. There is a wide range of chamber designs and sizes including walk in rooms that are capable of performing this test. Hot, humid air is created by bubbling compressed air through bubble (humidifying) tower containing hot deionized water.

Salt solution is typically moved from a reservoir through a filter to the nozzle by a gravity-feed system. When the hot, humid air and the salt solution mix at the nozzle, it is atomized into a corrosive fog. This creates a 100% relative humidity condition in the exposure zone. For a low humidity state in the exposure zone of the chamber, air is forced into the exposure zone via a blower motor that directs air over the energized chamber heaters.

Applications of salt spray testing:

- Salt spray test is a tool for evaluating the uniformity of thickness and degree of porosity of metallic and nonmetallic protective coatings.
- The test is useful for evaluating different lots of the same product, and it's especially helpful as a screening test for revealing a particularly inferior coating.
- Salt spray tests have been implemented to test the resistance of aluminum alloys to exfoliation corrosion.
- The salt spray test is considered to be most useful as an accelerated laboratory corrosion test that simulates the effect of marine atmospheres on different metals, with or without protective coatings.

Types of salt spray tests

- The neutral salt spray test: It's the most commonly used salt spray test in existence for testing inorganic and organic coatings, especially where such tests are used for material or product specifications. The duration of this test can range from 8 up to 3000 h. depending on product or type of coating.
- The acetic acid – salt spray test: It's used for testing inorganic and organic coatings, but is particularly applicable to the study or testing of decorative chromium plate plating and cadmium plating on steel or zinc die-castings and for the evaluation of the quality of a product.

- The copper–accelerated acetic acid-salt spray test : is primarily used for the rapid testing of decorative copper-nickel -Chromium or nickel-chromium plating on steel and zinc die-castings. It is also useful in the testing of anodized, chromate, or phosphate aluminum.
- Types of salt spray cabinets
Its have range in size from extremely small bench–top cabinets to large walk–in types. The small bench–top models are not practical; they have been found to be difficult to control and should be avoided. The large walk–in types have been developed to be controlled capably, but they are very expensive. The most commonly used cabinet is the top opening type. [28]

High Temperature Corrosion Testing:

- Autoclave corrosion tests are a convenient means for laboratory simulation of many service environments. The reason for such tests is to recreate the high temperatures (HT) and high pressures (HP) commonly occurring in commercial or industrial processes.
- Factors affecting corrosion behavior are often intimately linked to the conditions of total system pressure, partial pressures of various soluble gaseous constituents and temperature. There are many HT/HP environments of commercial interest, which include those in industries such as petroleum, nuclear, chemicals, aerospace and transportation where reliability, serviceability and concerns for corrosion are paramount.
- Corrosion coupons can be placed in the aqueous phase, vapor space, or at phase interfaces depending on the specific interest involved. Additionally, it is also possible to conduct electrochemical tests in HT/HP vessels. If multiple liquid phases are present it can be necessary to stir or agitate the media or test vessel to produce mixing and conditions whereby the corrosion test specimens are contacted by all of the phases present.
- Special magnetic and mechanical stirrers are available that can be used to produce movement of the fluid to produce a mixing of the phases. In some cases where contact of the specimens with both liquid and gaseous phases is important in the corrosion process, it may be necessary to slowly rotate or rock the test vessel to produce the intended results. HT/HP corrosion tests have special requirements not common to conventional corrosion experiments conducted in laboratory glassware.

- Four variations of common HT/HP test methods that have been found to be useful in materials evaluation involving corrosion phenomena and these include:
 - Windowed test vessels:
special transparent windows and other fixtures have been used to make visual measurements or observations within the confines of test vessels. Besides being able to withstand the pressures, temperatures, and corrosion environments, these windows may have to perform other functions related to the introduction of light or other radiation if these are part of the test variables.
 - Electrochemical measurements:
most conventional electrochemical techniques have been used for experiments conducted inside HT/HP vessels. The most critical electrochemical component in these experiments always has been the reference electrode.
The design and construction of the reference electrode is particularly important, as it must provide a stable and standard reference potential. In many applications, test vessels have been modified to accommodate an external reference electrode to minimize the effects of temperature, pressure, or contamination, or a combination thereof.
 - Hydrogen permeation:
hydrogen charging is often a problem that affects materials submitted to HT/HP test conditions. In such cases, it may be necessary to measure hydrogen permeation rates and diffusion constants in order to estimate the potential hazard of hydrogen attack. For hydrogen permeation measurements at high temperatures, it may be imperative to use solid state devices.
 - Mechanical property testing:
HT/HP vessels have been designed to conduct a variety of mechanical tests such as slow strain rate (SSR), fracture or fatigue testing. The main problem is always one of selecting the fixtures that could withstand the corrosive environments generated in HT/HP tests. [27]

3. Experimental part

3.1 Program of tests

I used coating which are studied at Department of Material Science of Technical University of Liberec and coating methods according to equipments that available for these methods in this department.

For porosity tests

- Determination of porosity of protective coating on steel (Czech standard ČSN 038154). Method after this standard is not using electrical current
- Determination of porosity test for the protective paint coating after (Czech standard ČSN 673084). Method B, i.e. electrographic method.

For corrosion test.

- Metallic and other inorganic coatings. Test by using sulfur dioxide SO₂ with general condensation of moisture (Czech standard ČSN ISO 6988).
- Cleaning specimens from corrosion product after corrosion test (Czech standard ČSN ISO 8407).

3.2 Preparation of samples

Degreasing the panels by using ultrasound device with using acetone for three minutes then I use distilled water for one minute and finally with using alcohol three minutes after that we dry the panels by using drier machine then it will be ready for coating but with out holding the panels with hand.

I used many types for coating the panels as the following:

- Unmodified polyethylene RM 8343.
- Modified polyethylene E5000 (2040219/03).
- Modified polyethylene RM 8343 (1030722/11).
- Marwin -TiN.
- Marwin - G
- Marwin - SI
- MT
- PT
- TiO_x
- Zincethylsilicate paint

- For coating the panels there are many methods:
 - Using the electrical furnace.
 - Using electrical heating gun.
 - Using PVD coating method.
 - Spray painting.

For samples prepared at our department was used the sheet from low-carbon Czech steel 11 321.21 after standard ČSN 411321 (which is usually used for cold forming) with thickness 1 mm. For samples prepared at SHM Ltd. Šumperk was used sheet with thickness 2 mm, because the deposition was made at higher temperatures, at which the deformation of thin plates can occur. The material used for this purpose was carbon steel with about 0,5 % of carbon (Czech steel 12050 after ČSN 412050).

3.2.1 PE coating.

Polyethylene is coating suitable for service temperatures up to 60°+80°C depending on the type of materials used (LDPE –MDPE-HDPE). Typical coating thickness is from 1, 2 mm to 3 mm. [13]

I used two methods for coating the panel with polyethylene by electrical furnace and electrical heating gun.

- For the electrical furnace I used 15 panels and I covered 9 of them with unmodified polyethylene RM 8343, and another 3 of them I covered with modified polyethylene E5000, and the last 3 panels I covered with modified polyethylene RM 8343, after that I place them on a sheet of metal and put them in the furnace on temperature 220°C for 30 minutes until the powder be melted on the panels but after 15 minutes I checked them and the powder was melted but the initiated layers was not uniform, so I put them again to the furnace for more 15 minutes but with out any change in the layer were the layer still not uniform. So I canceled them. This process seemed to give unsatisfactory quality of coatings. The coatings prepared in this way were non uniform in thickness, with rough pores and uncoated places. For this reason was tried other method of coating.
- For the second process of the coating by using electrical heating gun, I mark some panels with letters by the marker after us degreasing them. I start with letters as the following:
 - For panel C I covered with polyethylene unmodified powder

using cold panel and puffing the powder with spoon and glutting it with cylindrical glass bar and heated with the heating gun from the lower surface of panel after it melt, we repeat the process by adding some powder and the result for the initiated layer was good.

- For Panel E I covered with the same type of powder but with using preheated panel and poring with powder by knocking on the spoon plus glutting by the cylindrical glass bar and after that we preheat the panel with powder to melt it but the result was not good because the coating was delaminated (it's take off from panel), so I canceled this panel.
- For panel Panel A I covered with Borecene RM 8343 modified powder by using cold panel and puffing the powder with spoon and glutting it with cylindrical glass bar and heated with the heating gun from the lower surface of panel after it melt. I repeat the process by adding some powder and the result for the initiated layer was good (uniform) after that I measured the thickness of the layer it was $135 \pm 20 \mu\text{m}$.
- For Panel B I covered with the same process of panel A. Where the result was the same but the layer was less uniform than A where the thickness was $103 \pm 50 \mu\text{m}$.
- For Panel D I covered with the same process of panel B. Where the result was uniform with thickness $122 \pm 40 \mu\text{m}$ this panel is holed from against side of the hole of the same panel A.
- For Panel F I covered with modified powder E5000 by apply the same process of the last three panels but it's different from type of powder where this type is more finer that the last one in addition of that it's doesn't melt quickly and the initiated layer is not uniform and there are small bubbles appears under the layer of coating. The thickness was measured and it's was $97 \pm 40 \mu\text{m}$.
- For Panel G I covered with E5000 powder with out adding additional layer, the initiated layer is not uniform and there are small bubbles appears under the layer of coating. The thickness was $373 \pm 70 \mu\text{m}$.

- For Panel H I covered with E5000 powder with out adding additional layer, the initiated surface layer is very uniform appears of small bubbles under the layer of coating. The thickness was measured and it's was 431 +/- 100 μm . Type of powder that used in the panel G and H is the same of panel F.

3.2.2 TiN and similar coating.

For the TiN and similar was used the PVD coating method. (made in SHM)

- PVD technology is based on physical principles, evaporation or sputtering of materials included in a coating (eg. Ti, Al, Si, Cr,) and their subsequent deposition on sample. The material is evaporated and concurrently ionized by the arc from electrodes. The ionized material (eg. Ti^+ , Ti^{2+} , etc.) is accelerated towards the substrates by negative bias, which is applied to them. While traveling it ionizes atoms of gas atmosphere (eg. N_2 , Ar,) as well. The ionized atoms create the actual deposited coating by surface reaction when reaching the substrate. [29]
- For the PVD method we used the following panels
 - For panel (0405-0255) I covered with MARWIN TiN coating, its applied on carbon steel, the characteristics are a double layered system consisting of TiN base layer with a minimum Al content and pure TiN top layer, and application for this type is use for less intensive cutting applications, low cutting speeds, and the properties of this coating is 25 Gpa for micro hardness, 2-3 μm for thickness, 0.008-0.12 μm for roughness and 550°C for thermal stability.
 - For Panel (0410-0005) I covered with MARWIN-G coating, it applied on carbon steel, the characteristics of this type is TiAlN monolayered system consisting of a layer with evenly changed stiochiometry, where this type of coating is use for cast iron and hardened steel machining. The basic properties of the coating is 33 Gpa for microhardness, thickness is 2-3 μm , roughness 0.10-0.13 μm and the thermal stability upto 900 °C.
 - For Panel (0405-0199) I covered with MARWIN-SI coating, its applied on carbon steel, the characteristics of this type is AlTiSiN nano-composite system consisting of base layer with high hardness and top layer with high thermal and chemical stability, where this type of coating is use for

all purpose coating with wide scale of application for machining as well as for cutting and molding. The basic properties of the coating is 43 Gpa for microhardness, 2-3 μm for thickness, 0.10-0.15 μm for roughness and $> 1000^\circ\text{C}$ for the thermal stability.

- For Panel (0404-0201) I covered with MARWIN-PT coating, it applied on carbon steel, the characteristics of this type it has dark color, where the layer thickness is 4.69 μm , with roughness 0.25-3 its applied on carbon steel.63 μm , the microhardness is 41.6 Gpa.
- For Panel (0403-0263) I covered with MARWIN-MT coating, it applied on carbon steel, the characteristics of this type it has gold color, where the layer thickness is 5.5 μm , with roughness 0.35-3.8 μm and the microhardness is 43 Gpa.

3.2.3 TiOx coating.

The samles of TiOx are coated by Planar RF Reactor at Laboratory of Plasma Technology at Department of Material Science in Technical University of Liberec the method that used for coating the samples is PECVD (plasma enhanced chemical vapour deposition), where the working parameters are pressure: 6 Pa, source input: 30 W, time: 2 hours, bias: 350 V, the gas mixture ratio for depositing was (20 sccm) argon to (2 sccm) titanium isopropoxide where (sccm = standard cubic centimeter). The samples were deposeted on four different temperatures 100°C , 200°C , 300°C , 400°C and the thickness of each samples were 2.1 μm for 100°C , 1.5 μm for 200°C , 1.4 μm for 300°C , 2.2 μm for 400°C . The substrate of basic metal for the samples was forged low carbon steel with 1mm thickness and surface area of 20 cm^2 , [30]

The description of the PECVD is the component elements of the film to be deposited are introduced into reaction chamber via gaseous precursor reactants. The reactants are mass-transported to the surface of the hot substrate, where they are adsorbed. If the substrate is heated to an appropriate temperature for the desired chemical reaction to take place, films of the material to be deposited form on the substrate surface while reaction by products are pumped out of the system . [31]

3.2.4 Paint – Pragokor Metal E.

For paint panel is two component, modified and solvent based zincethylsilicate paint, with high content of metallic zinc, its prosperities is inorganic coating with temperature resistance up to 400°C as a single coat and that be even enhanced by an aluminum pigmented silicone top coat, long term corrosion protection (at least 15 years) in very aggressive atmospheres without any maintenance, we can raise the life time by top coating with organic paints like epoxy, coal tar epoxy, polyurethane. It can be used as single coat or top coated primer for long term steel structures protection in aggressive atmospheres or other environments pH value with in 5.5-10.5 (sea water), the structures exposed to high humidity or water spraying. The thickness of applied coating is 75 µm, and the substrate metal thickness 1mm with surface area 15x 10 cm²., (specimen made in Pragochema Praha). [15]

3.3 Testing methods

3.3.1 Porosity tests

Porosity test after (Czech standard 038154 ČSN).

The description of porosity test for ČSN 038154 on steel is the following:

I dissolved 5g of sodium sulfate Na₂SO₄ and 1g of potassium ferricyanide K₃[Fe(CN)₆] in about 800 ml of distilled water and after dissolution I completed them by water adding to 1 liter of solution. The filtration paper is soaking 1 minute in solution after I cut around the panel. Then the access solution is drop down then it attached to surface area of all surface, if air bubbles is appears under the paper it must be removed by pressing on it by cylindrical bar.

The duration of test is 10 minutes then the paper is removed from solution and dried between two filtration papers and then we wash it by distilled water and let the paper to dry in normal room temperature.

After drying a number of blue green stains (spots) will appears on paper and I collect them and divide them on the surface area of panel.

Porosity test after (Czech standard ČSN 67384 - method B).

The description of the porosity test of paint protective coating for 673084 ČSN as the following:

In this test I have two types of solutions; the first one is to mix 50 g of potassium nitrate KNO_3 with 1 liter of distilled water and filled in bottle and this solution is used for soaking of photo paper. Where the second solution is to mix 25 g potassium ferrocyanide $\text{K}_4[\text{Fe}(\text{CN})_6]$ in 1 liter of distilled water and filled in bottle and this solution is used for developing the blue stains. After I prepare the solutions for test I soaking the photo paper in potassium potassium nitrate KNO_3 for 15 minutes then I but the paper on the coating panel on the glossily side after I but the panel on a sheet of aluminum after that I but a sheet of aluminum and I but a weight of 1 kilogram and then I bring the source of direct current and connect it and set it at 6V then I connect the positive port to aluminum sheet under the panel and the negative port to aluminum sheet upper the photo paper and connect the button for converting the reading on screen from voltage to ampere and I wait for 20 seconds the duration of test. After that I turn off the source of direct current and removed the negative and positive port then I take off photo paper from the panel and soaking in potassium ferrocyanide $\text{K}_4[\text{Fe}(\text{CN})_6]$ for 30 minutes for develop the blue stains, after that I rinsed the paper with distilled water and left dry, the result will be the number of stains that appears on paper divided by the surface area of the panel in ($-\text{/cm}^2$).

3.3.2 Corrosion test.

For the corrosion test (Czech standard ČSN ISO 6988).

I have two ways continuous test and test in cycle I recommend to make test in cycle of 24 hours where 8 hours in chamber and 16 hours out side the chamber in the air.

I can get sulfur dioxide SO_2 from the following chemical compound: 202.5 g of sodium sulfite $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ / 1 l of distilled water and 200 ml about 83% of sulfuric acid H_2SO_4 /1 l of distilled water, I get the amount of sulfur dioxide SO_2 by mixing of 10 ml of sodium sulfate with 10 ml of sulfuric acid to get the requirement of SO_2 for our humidity chamber of size 270 dm^3 . The temperature of test $40^\circ\text{C} \pm 3^\circ\text{C}$ and recommended heat input is 1 kW.

I cleaned the samples by using acetone before the test and covered the uncoated side by adhesive tape. The samples then are butted in chamber and the distance between 20 mm between each other and the distance between the wall and sample must be more than 100 mm the distance between edge of sample and the water level must be 200 mm, where the amount of water must be $2 \text{ l} \pm 200 \text{ ml}$. The sample can not be placed under the another sample.

The material is used to hang the sample must be from nylon fiber. The required purity of distilled water is given by its maximum allowed electrical conductivity, i.e. $500 \mu\text{S} \cdot \text{cm}$. In the chamber we put 0.2 of sulfur dioxide SO_2 . And the regulator electrical heater must be on level 40°C . The testing cycle is 24 hours where 8 hours in chambers and the rest 16 hours will be out the humidity chambers, the water of the chambers must be changed every cycle and also sulfur dioxide and the chambers must be opened during draining. The temperature of the room must be $23^\circ\text{C} \pm 5^\circ\text{C}$. The relative humidity must not be more than 75%. After finishing the test, the samples are extracted from the chamber but before that I have to hang the samples in room to remain SO_2 from the samples to see the corrosion occurs.

For cleaning specimens from corrosion product after corrosion test (ČSN ISO 8407)

For removal corrosion product from the specimens I used about 10 minutes-pickling using solution C.3.5 after (ČSN ISO 8407) by dissolving solution of 500 ml HCl (hydrochloric acid with density $1,19 \text{ g/ml}$) with 3,5 g of hexamethylenetetramine (urotropine) in 1 l of distilled water at room temperature. As-pickled specimens were rinsed by water then by denaturated ethanol and dried by hot air machine. [32]

3.4 Results of tests.

3.4.1 Results for the porosity test.

Results for the porosity test of protective coating on steel for (Czech standard ČSN 038154) I have the following results.

Table 6 : Results of porosity test after Czech standard ČSN 038154

No	Samples	Description	Number of spots per 20 cm ² [-]
1	AQ002 TiOx 200°C	Fine and small spots diameter	50
2	AQ005 TiOx 300°C	Fine and small spots diameter	36
3	AQ007 TiOx 400°C	Fine and small spots diameter	175
4	AQ011 TiOx 100°C	Fine and small spots diameter	150
5	405 – 255 Marwin-TiN	Small and large spots diameter	140
6	410 – 005 Marwin G	Small and large spots diameter	115
7	405 – 199 Marwin-Si	Small and large spots diameter	52
8	405 – 263 MT	Small and large spots diameter	80
9	404 – 201 PT	Fine and small spots diameter	110

- For the porosity test of protective paint coating after Czech standard ČSN 673084 method B, i.e. electrographic method, the results was the same for all the samples because of high sensitive of this test to the coating types that we used in test.
- For the porosity test of protective paint coating on steel by using source of direct current after ČSN 673084 - method B, the results were the same for all the samples because of high sensitivity of this test to the coatings that we used in test.

3.4.2 Results for the corrosion test.

For the corrosion test after (Czech standard ČSN ISO 6988), I make nine cycles during nine days as the following where each table is result of one

Table 7: Results for humidity test for cycle number 1

No	Sample	Desecration
1	Uncoated 13.155g	The surface is all corroded; we can measure the weight to know the corrosion mass defect.
2	Marwin-SI	The original color was grey and it's change to darker grey with initiating two stains one darker and one lighter with diameter of 2 x 5 mm.
3	Uncoated thick	5 circular red brown stains with diameter of 2 mm, small pits in upper side, and dark band on 2 mm from periphery.
4	D polyethylene	Initiating air bubbles in the lower part with accents 6 x 25 mm.
5	TiOx 400°C	Very fine pitting in right vertical half and the pitting with shade brown.
6	TiOx 300°C	Finest pitting from the lower side at right corner and at middle from the left side (hardly seen), the accents of pits around 5 x 8 mm.
7	TiOx 200°C	No changing is appears (no results).
8	Marwin- TiN	Original color was gold color and it's nearly removed, irregular distribute red brown stains mainly down and up, number of stains around 15 with accents 2 x 2 mm.
9	Pragokor Metal E	The original color is grey and from the first side some area is changed to darker grey with very fine stains, the second side the same of the first but with less accents
10	Uncoated Thin	Uniform brown black appears
11	PT	Original color was gold is changed to very fine brown pitting, pitting nearly uniform, greater brown stains in the left 5x4 to 5x2.

12	MT	Original color was gold color and it's nearly removed, irregular distribute red brown stains mainly down and up, number of stains.
13	B polyethylene	Two stains of air initiated under the layer in lower third and in the left upper corner.
14	Marwin-G	Original color was grey black and its change to darker grey and initiated a number of stains about 15 grey brown with accents of 2 x 2 mm mainly on periphery.
15	TiO _x 100°C	No change is appears (no result)
16	A polyethylene	Two stains or air is initiated under layer, one stain in the upper side and the other in the right lower side with accents 3/5 from the all surface area.

Table 8: Results for the humidity test for cycle number 2

No	Sample	Deseccration
1	Uncoated 13.155g	Corrode all surface with appearance of red Brown stains with accents of 4x6 mm, we can Measure the weight to know the mass defect.
2	Marwin-SI	The darker grey color is changed to brown color and initiating of red stain in the right mid of sample with around 17 pits and they have different diameter from 1-3 mm, and the lighter dark grey is supply around the darker grey stains near to the periphery.
3	Uncoated thick	Uniform brown black appearance with initiate of 5 pits of brown black wit diameter of 3mm in the upper and lower side.
4	D polyethylene	Increasing the air bubbles with accents of 8x30 mm in lower side and initiate of brown surface under the layer in the lower corners and in the upper right corner.
5	TiO _x 400°C	Increasing of very fine pitting in the right vertical half to be 60% of all area, the pitting is shade brown, with initiate of black brown stains in the right lower side with accents 3x4mm.

6	TiOx 300°C	Increasing of finest pitting from lower side with accents 1/3 of all area.
7	TiOx 200°C	No change is appears (no results)
8	Marwin- TiN	The original gold color is start back again after the surface layer of corrosion is start to remove with increasing of circular stain diameter to be 4 mm and with central red green of 2 mm.
9	Pragokor Metal E	Increasing the darker area of the two side with initiating of different spot diameter in the upper side about 3 mm and its decrease in lower side about 1 mm.
10	Uncoated Thin	Increasing the red black spot.
11	PT	Uniform brown spots like the first cycle.
12	MT	Brown red in the mid from upper to lower side and light brown red from left side in vertical from lower to upper with increase of pits with accents 4 mm in the right and in the mid .
13	B polyethylene	Increasing the air bubble to 90% of the all area with initiate of corrosion under layer in the left lower corner.
14	Marwin-G	The darker grey is removed to brown red and initiate of some pits in all area with accents 3 mm.
15	TiOx100°C	Initiating of finest pitting around the periphery of the panel exactly in the upper side with accents half of all area.
16	A Polyethylene	Increasing the air bubble under layer to 80% of area from lower to upper side with initiate of corrosion in the mid of panel with accents of 5x25 mm and in the lower side about 2x20mm to right side.

Table 9: Results for the humidity test for cycle number 3

No	Sample	Desecration
1	Uncoated 13.155g	Appearance of fine pitting in the upper mid side.
2	Marwin-SI	Changing the color to darker black brown with increase of the circular pits diameter to be 3 mm.
3	Uncoated thick	Increasing the corrosion rate exactly the black area and it's supply to many branches with appearance of red spot in the lower side with accents 10x7 mm.
4	D polyethylene	Increasing the brown area under layer exactly in right upper side and in lower left corner.
5	TiOx 400°C	Increasing the pits size with appearance of some stain light brown in the right upper side about 6 stains with accents of 2x3 mm.
6	TiOx 300°C	Increasing of fine pits to be around half of all area.
7	TiO ₂ 200°C	Appears of some scratch in lower mid side and appearance also of finest pits in the upper right corners and in the lower side in the mid near the periphery.
8	Marwin-TiN	Little change in the original color to be darker near to light brown.
9	Pragokor Metal E	Appears of some fine pores in front face all area and around the periphery, and the back face no new change.
10	Uncoated Thin	Appearance of red pores in some area, brown stains in all area and black stains in the mid.
11	PT	Increasing of spot size and appearance of red spots.
12	MT	Changing the color to darker with increase of spot size, in some place the coated is removed in the left vertical side and in some other place the coating is start to remove.
13	B polyethylene	No thing is change
14	Marwin-G	Changing to darker color than the last cycle. With increase of pore and the coating is removed in neiberhood.
15	TiOx 100°C	Increase of brown black color and size.
16	A polyethylene	Air bubble is increased with initiate of black spot on the left mid side near the periphery with accents of 2x3 mm, 3x3 mm.

Table 10: Results for the humidity test for cycle number 4

No	Sample	Desecration
1	Uncoated 13.155g	Increase of spot size in vertical right side.
2	Marwin-SI	The same of last cycle (no change).
3	Uncoated thick	The black color removed and dark brown color increased and initiated some fine pores and the circular spots start removed.
4	D polyethylene	Air bubble increased and the brown area under layer is also increased exactly in left side on periphery and in the right side on the periphery and in lower corners.
5	TiO ₂ 400°C	Initiate fine pores with increase of light brown stains size with appearance of red stain with accents 3x6 mm in the upper left side near to periphery.
6	TiO _x 300°C	Initiate of some fine pores with increase of light brown stains size.
7	TiO _x 200°C	Initiate of some fine pores in lower right corner and increase the area of finest pits in the mid of the left side with increase of some scratch size in the mid lower side.
8	Marwin-TiN	Increase the circular spot diameter with initiate of some circular small spot in the light brown area.
9	Pragokor Metal E	Increase the number of pores with appearance of some pores in the back side, initiate of some circular light grey stains in the back side.
10	Uncoated Thin	Increase of brown color area with decreasing black color area, with increase also number of red circular spot with diameter of 1mm.
11	PT	Increase the dark brown color area with initiate of small pores in all area.
12	MT	Increase the dark brown color area with increase of size spot with decrease of light brown color area.
13	B polyethylene	The layer of coating is start delaminating, increase of brown color around the periphery of the sample.
14	Marwin-G	The dark brown color is converted to small spot with increase of circular light spot size.
15	TiO _x 100°C	The black color is removed and the light brown color increased.
16	A polyethylene	The layer of coating is start delaminating with increase of brown color on the periphery around the sample.

Table 11: Results for the humidity test for cycle number 5

No	Sample	Desecration
1	Uncoated 13.155g	The light red spot in the lower right corner is convert to brown with initiate of some pores and also the brown stains is converted to light brown.
2	Marwin-SI	The red pore is converted to light brown and some of them removed.
3	Uncoated thick	The dark brown increased and converted to small irregular contact shape.
4	D polyethylene	The light grey area is increased little, also the brown color under layer is increased around the sample and the pores exactly in left and lower right increased.
5	TiOx 400°C	The stains increased in size on all area.
6	TiOx 300°C	Initiated of burble color with increased in stain size of light brown and decreased the dark brown area.
7	TiOx 200°C	The finest pits increased on some area in upper corners and left, right near the periphery and in lower right corners with increased of scratch area.
8	Marwin- TiN	The light color is converted to little dark with increased of circular and irregular stains size in all area.
9	Pragokor Metal E	The numbers and size of stains is increased, initiated some of branches (like small crack) in all stains in front and back side exactly in front side.
10	Uncoated Thin	The dark brown area is removed from the mid area, the light red stains increased in size in lower side with accents of 10x25 mm.
11	PT	The red pores converted to brown and the dark brown color is increased.
12	MT	Initiated in the pores small crack (branch), the light brown area and the dark brown increased, ad the stains is start to removed.
13	B polyethylene	The brown color under layer increased around the periphery of sample.
14	Marwin-G	The brown color is converted to small stains, the circular spot is start to removed and the dark brown color in lower side increased also.
15	TiOx100°C	Some pores color is converted from light red to red with increased of dark brown color size and the black color area decreased.
16	A polyethylene	The brown color area under layer increased little pits, also pores size increased in left side on periphery.

Table 12: Results for the humidity test for cycle number 6

No	Sample	Desecration
1	Uncoated 13.155g	Pores increased in number and size in same area.
2	Marwin-SI	The burble color removed to be mostly black color with pores of light brown color.
3	Uncoated thick	Nothing is change.
4	D polyethylene	The brown area under layer increased in left and right side of sample.
5	TiOx 400°C	The light brown color area increased and appears burble color in left upper side with accents 6x25 mm.
6	TiOx 300°C	Nothing is change.
7	TiOx 200°C	Initiated of some irregular stains with light brown color with appears of burble color in upper corners.
8	Marwin- TiN	Disappears of circular spot and initiated some light brown pores in lower side near the periphery.
9	Pragokor Metal E	The number of spots increased in two sides and the crack inside the spots also increased little pit.
10	Uncoated Thin	Initiated of some pores in many area with change of brown color to burble and brown.
11	PT	Nothing is change.
12	MT	The color of surface is changed to dark brown, and the stains color is changed to dark brown also.
13	B polyethylene	Nothing is change.
14	Marwin-G	The surface color is changed to dark burble and the spots are removed.
15	TiOx100°C	Brown stains are initiated in upper mid side.
16	A polyethylene	The layer of coating is delaminated completely.

Table 13: Results for the humidity test for cycle number 7

No	Sample	Desecration
1	Uncoated 13.155g	Appears of black color area with initiated a lot of pores in back side.
2	Marwin-SI	The color of pores is converted to brown with removed some of them and initiated of one red spot with accents 1 mm in upper right side.
3	Uncoated thick	Initiated of 20 red spots with different diameter in all area.
4	D polyethylene	Increased the brown color under layer little pit in all sides.
5	TiOx 400°C	Initiated of small black pores in vertical right side, also the size of pores increased in lower right corner.
6	TiOx 300°C	The burble color is changed to brown with initiated of 18 red spots in all area with different diameter from 0.5-2 mm.
7	TiO ₂ 200°C	The burble color is increased in all area, the brown and black stains also increased.
8	Marwin- TiN	Initiated in the black spots brown spots with initiated in the upper corners light brown spots.
9	Pragokor Metal E	Initiated of white fine pores in the middle of front side and also little in the back side.
10	Uncoated Thin	Appears dark brown stain and concentrate in the mid of sample, with little burble color on the periphery around the sample.
11	PT	The pores diameter is increased little and the color of middle area is changed to light brown.
12	MT	Appears of big light brown stains in middle, vertical red stain appears in the upper left side with accents 5x30 mm.
13	B polyethylene	The layer of coating is delaminated from the lower right corner.
14	Marwin-G	The surface color is changed to dark brown with initiated of new 5 pores in lower left corner, in mid right side and in mid left side with accents 2 mm.
15	TiOx100°C	Initiated of 10 red spots in different location and size in middle and lower side.
16	A polyethylene	The sample is oxidized with initiated of black color line near the periphery in upper, lower and right side.

Table 14: Results for the humidity test for cycle number 8

No	Sample	Desecration
1	Uncoated 13.155g	Appears of fine pitting in the mid and lower of the back surface and finest pores in front side in middle of sample.
2	Marwin-SI	The red spots are removed.
3	Uncoated thick	Most of the red spots are converted to brown color.
4	D polyethylene	The layer of coating delaminated from lower right corner.
5	TiOx 400°C	Most of black pores are removed and initiating of light brown area in right vertical side.
6	TiOx 300°C	Some of red pores removed by converted to brown color.
7	TiOx 200°C	The light brown stain increased in all area with decreasing in black stains and burble stain.
8	Marwin-TiN	Appears of light burble color, black spot in upper left side and some pores in upper side near the periphery.
9	Pragokor Metal E	Converting the fine cracks in to irregular small spots light grey color and its concentrating in back side than the front side.
10	Uncoated Thin	Appears of light red pores in the mid side with different size.
11	PT	Nothing is change
12	MT	The dark brown color is change to brown with appears of dark brown circular and irregular spots with large number and different size.
13	B polyethylene	The coating layer is delaminated completely with appears of brown line on surface around the periphery of sample.
14	Marwin-G	The pores color is changed to dark brown.
15	TiOx100°C	The size of pores increased.
16	A polyethylene	The surface of sample is completely corroded to black color.

Table 15: Results for the humidity test for cycle number 9

No	Sample	Desecration
1	Uncoated 13.155g	Nothing change
2	Marwin-SI	Most of the surface layer color is changed to light brown.
3	Uncoated thick	The color of the most spots is changed to dark brown.
4	D polyethylene	The delaminating of coating layer increased in lower side, the brown line color under layer around the sample near the periphery increased.
5	TiOx 400°C	The light red pitting increased, with initiated of light brown color in vertical mid area.
6	TiOx 300°C	Most of the surface color changed to dark brown and black color.
7	TiOx 200°C	Fine irregular pores initiated in upper right corner and in middle, and in lower left corner around the black stains with burble color.
8	Marwin-TiN	Appears of light brown spots in lower side, also brown irregular and circular spots in middle and in upper side near the periphery.
9	Pragokor Metal E	The light grey fine pits increased in number and diameter in front side, also the brown pores increased in the front and back sides around the periphery, and in lower and mid of front side.
10	Uncoated Thin	The red pores increased in number and size in all surface area, appears of burble color in all surface area.
11	PT	The color is changed to dark brown.
12	MT	The color is changed little pits to brown color in all surface area.
13	B polyethylene	After the coating layer delaminated the brown and black color increased around the sample near the periphery.
14	Marwin-G	Nothing change
15	TiOx100°C	The pores color is changed to dark brown in all surface area.
16	A polyethylene	Initiated of some light brown pits in upper and in lower side after delaminated of layer.

Table16: Summary of last nine cycles of corrosion test.

samples		Cycle1	Cycle2	Cycle3	Cycle4	Cycle9
1	Uncoated 13.155g	Corrosion traces				
2	Marwin-SI		Corrosion traces			
3	Uncoated thick	Corrosion traces				
4	D Polyethylene		Corrosion traces under layer			Start delamination of layer
5	TiOx 400°C	Corrosion traces				
6	TiOx 300°C	Corrosion traces				
7	TiOx 200°C				Corrosion traces	
8	Marwin-TiN	Corrosion traces				
9	Pragokor Metal E			Corrosion traces		
10	Uncoated thin	Corrosion traces				
11	PT	Corrosion traces				
12	MT	Corrosion traces				
13	A Polyethylene		Corrosion traces under layer		Start delaminat- ion of layer	
14	Marwin-G	Corrosion traces				
15	TiOx100°C			Corrosion traces		
16	B Polyethylene		Corrosion traces under layer		Start delaminat- ion of layer	

Table17: Results after removal of corrosion product

No	Sample	Desecration
1	Marwin-TiN	The rust is removed from the coating layer except small amount in the upper right corner and in lower left side near periphery; the coating layer is removed completely from the specimen.
2	Marwin-SI	The rust is removed from coating layer completely with removing some area of coating with accents of 60%.
3	PT	The rust is completely removed and the coating layer is removed also about 40% from all area.
4	MT	The rust is completely removed with removing coating layer from some area in the lower left corner and in upper left side.
5	Marwin-G	The rust is removed completely with removing coating layer from the all surface area about 35%
6	TiOx 300°C	The rust is removed completely from the coating layer with removing the coating layer almost in all area.
7	TiOx 200°C	The rust is completely removed with removing little pits from coating layer exactly in right upper and lower corners with accents 6% from all area.
8	TiOx 400°C	The rust on the coating layer is removed completely except small area in the lower right corner the coating layer is also removed completely
9	TiOx100°C	The rust is removed and the coating layer is removed also little pits in the upper right corner
10	A polyethylene	The rust is removed completely.
11	D polyethylene	The layer of coating is delaminated completely with removing of 50% from initiated rust under layer .
12	B polyethylene	The rust is removed from under layer about 90% from all surface area.
13	Uncoated 13.155g	The rust is completely removed from the two sides except small area in lower right corner in front side.
14	Uncoated Thin	The rust is completely removed
15	Uncoated thick	The rust under layer is removed about 80% from all surface area.

For calculating the mass defect for the uncoated sample with weight 13,1559 g after removing corrosion product I have the following procedure:

Uncoated sheet thickness 1 mm from Czech steel 11 321 with dimensions 50x40 mm with original mass 13,1559 g after test and corrosion products removing 12,8242 so the corrosion mass defect was 0,3318 g.

Corrosion defect after 9 cycles (1 cycle consists from 8 hours in humidity chamber with SO₂ and 16 hours in room atmosphere) was 0,3318 g

onesided surface area= $5,4 = 20 \text{ cm}^2$

total surface area= 40 cm^2

total test duration = $9,24 = 216 \text{ hours} = 9 \text{ days}$

corrosion rate = $0,3318 / (40,9) = 0,000922 \text{ g per cm}^2 \text{ per day}$

$9,22 \text{ mg per cm}^2 \text{ per day}$

4. Discussions of the experiment results.

For the porosity test (Czech standard ČSN.0318154).

The samples can be divided to three groups according to number and size of blue spots on the filtration papers. These three groups can be arranged them according to the following:

- Group of high number and large size of spots include; Marwin-TiN, Marwin -G, MT, Marwin -SI, TiOx 400°C, and these types of coatings easy to corrode because of availability of a lot of pores in the coating layer that can not prevent the corrosive environment from the basic material.
- Group of half quantity and medium size of spots, include; TiOx 400C, TiOx 100°C, PT, where these types of coatings have medium range of spots so it's not easy in same time not difficult for metal to be corrosive.
- Group of less number and smallest size of spots, they are; TiOx 200°C, TiOx 300°C, these types of coatings have lower range of spots, so it defaults for metal to be corrosive.

For the corrosion test after (Czech standard ČSN ISO 6988)

I have for the corrosion test 16 samples and I divide them in to five groups:

- Group of MARWIN coating and they are five samples, three of them have gold color; MARWIN-TiN, MARWIN-MT MARWIN-PT, and the last two samples of group is MARWIN-SI and it have dark blue, where the second one have dark grey and it MARWIN-G .In this sample which have gold color, the color of samples nearly removed from the first cycle. Where the dark grey color is also defect from the first cycle except the dark blue color it have small change by appears small light and dark stains and start to defect from the second cycle.

Group of TiO₂ coating and it consist of four samples and they; TiOx 100°C, TiOx 200°C, TiOx 300°C, TiOx 400°C. Where two samples was affected little from first cycle and the other two doesn't affect

and they are; TiOx 200°C, TiOx 100°C. For the TiOx 100°C start defect from the second cycle, and the TiO₂ 200°C start effected with fine scratch in the third cycle, and effected with light brown stains from cycle number 6.

- Group of polyethylene powder coating consisting of three samples of borecene RM 8343 with different thickness, and the samples are A, B, and D. For the sample A coating layer start to delaminated in the cycle number 4 and completely delaminated in the cycle number 6, where the coating layer of the sample B start to delaminated in the cycle number 7 and delaminated completely in the cycle number 8, and the layer coating of the sample D start to delaminated in the cycle number 8. All the last samples start to effect with initiating air bubble under layer before start to delaminate; the best of the last sample was sample D because it starts delaminated at cycle number 8. The different results between them even they are coated with same type of powder, for initiating of small air bubble between layer and panel during of melting powder on panel and that for the bad applying method of coating powder on panel. Because it must be applied by preheating the substrate (basic metal) and spraying the powder on substrate by airless spray guns.
- Group of uncoated samples and it consist of three sample; uncoated thin, uncoated thick, uncoated 13.155g. All the last samples were affected from the first cycle.
- Pragokor Metal E (zincethylsilicate) paint coating. This coating affected from the first cycle by appears of some light grey spots on layer surface and in the third cycle appears some fine pores. Where in the last cycle light grey fine pits was appeared.

From the all last discussion I distinguish the following;

For porosity test after (Czech standard 0318154 ČSN).

The best of coating material was TiOx 300°C, and TiOx 200°C

For corrosion test after (Czech standard ČSN ISO 6988).

- I can distinguish three types of coating to the amount of corrosion occurs during the nine cycles and they are; Polyethylene powder is the best type of coating, because the coating layer doesn't effected but delaminated from sample and

that back to bad way of applying powder on sample for coating because of initiating small air bubbles under layer during coating, where air bubbles help the layer to be delaminating from sample after of increasing the amount of air by heating the sample from the humidity chamber during test. Because we were having three samples of same types of polyethylene powder and the layers of sample delaminated in different number of cycles according to the amount of air consisting under layer, the best of the sample was number D because it starts delaminating in cycle number 9.

- Paint (Pragokor Metal E) coating it's just affected in some small area as small fine pores in front side and round the sample on the periphery, with appears of small spots with light grey color, and this back to the thickness of the coating its about 25 times than the TiO_x and TiN and similar.
- TiO_x 200°C coating was the best of metallic coating types because it was affected with corrosion exactly after the cycle number 4 where the most of other metallic coatings effected from the first cycle.

For cleaning the specimens from corrosion product (Czech standard ČSN ISO 8407)

For this procedure I used 15 specimens all of them are cleaned from the corrosion product by using of inhibited hydrochloric acid except the of polyethylene layer the corrosion product doesn't removed completely and that back to the time of the procedure is not enough to removing all corrosion product. The coating layer of most of the specimens is removed from the effecting of corrosion product and that was exactly as the following:

- Specimen Marwin-TiN the coating layer is completely removed.
- Specimen TiO_x 400°C the coating layer is completely removed.
- Specimen TiO_x 300°C the coating layer is almost removed from all area.
- Specimen Marwin-SI the coating layer is almost removed about 60% from all area.
- Specimen PT the coating layer is removed about 40%
- Specimen Marwin-G the coating layer is removed little pit from all area about 25% from all area.
- Specimens MT, TiO_x 100°C and TiO_x 200°C, the coating layers from the last specimens was affected little pits about 3-5 % from all area.

So from the all specimens I distinguish that the best coatings are TiO_x 200°C, TiO_x 100°C and MT.

5. Conclusions.

For the theoretical part .

- The most corrosion cause pipeline integrity degradation includes the following.
 - Uniform or general corrosion.
 - Pitting.
 - Crevice corrosion.
 - Intergranular corrosion.
 - Erosion corrosion.
 - Environment-induced cracking.
- Factors that may prevent or contribute to the initiation and attack on buried pipelines include the following.
 - Pipe coatings.
 - Cathodic protection.
 - Soil conditions.
 - Temperature.
 - Stresses (residual and others)
 - Pipe pressure.
 - Cyclic loading effects.
- The most effective pipeline coatings include the following.
 - For external pipeline coatings*
 - Fusion bounded epoxy was and is being used.
 - Asphalts tar were used; and in the 1970's to present day.
 - Polyethylene powder, tape and extruded polyethylene jacket material was also use from the early 1950's to the present day.
 - For internal pipeline coating*
 - Many types are used Cement mortar coating, nylon (thermoplastic) coating, tube (liquid or powder) coating, inner (liquid paint) coating.
 - Cement mortar coating is the best type of internal lining for steel and cast iron pipes and transporting drinkable water.

For experiment part I have the following points.

- For the porosity test.
 - Best coatings are TiOx 300°C and TiOx 200°C.
- For the corrosion test.
 - Before cleaning the specimens from corrosion product.
 - The best protective coating from all samples was polyethylene powder.
 - The best type of TiOx coatings was TiOx 200°C.

- The Pragokor Metal E paint coating it has good coating and that back to high thickness of coating comparing to lower thickness in metallic coating.

After cleaning the specimens from corrosion product.

- The hydrochloric acid with hexamethylenetetramine is good for removing corrosion product from the coating layer.
- The best coatings are TiOx 200°C, TiOx 100°C and MT.
- The worst coating types are Marwin- TiN and TiOx 400°C.

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7. Appendix



Fig 4: applying powder of polyethylene on panels



Fig 5: Rectifier device

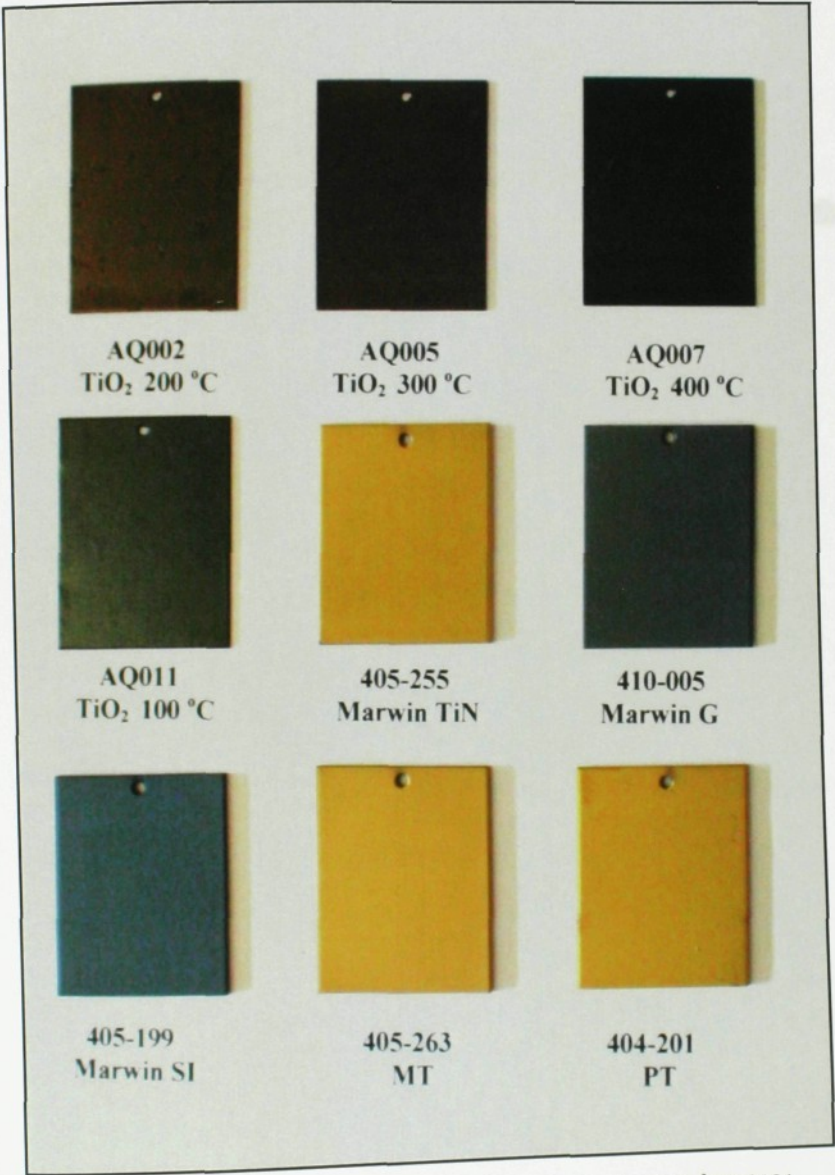


Fig 6: Types of sample used in porosity test after Czech standard ČSN 0381

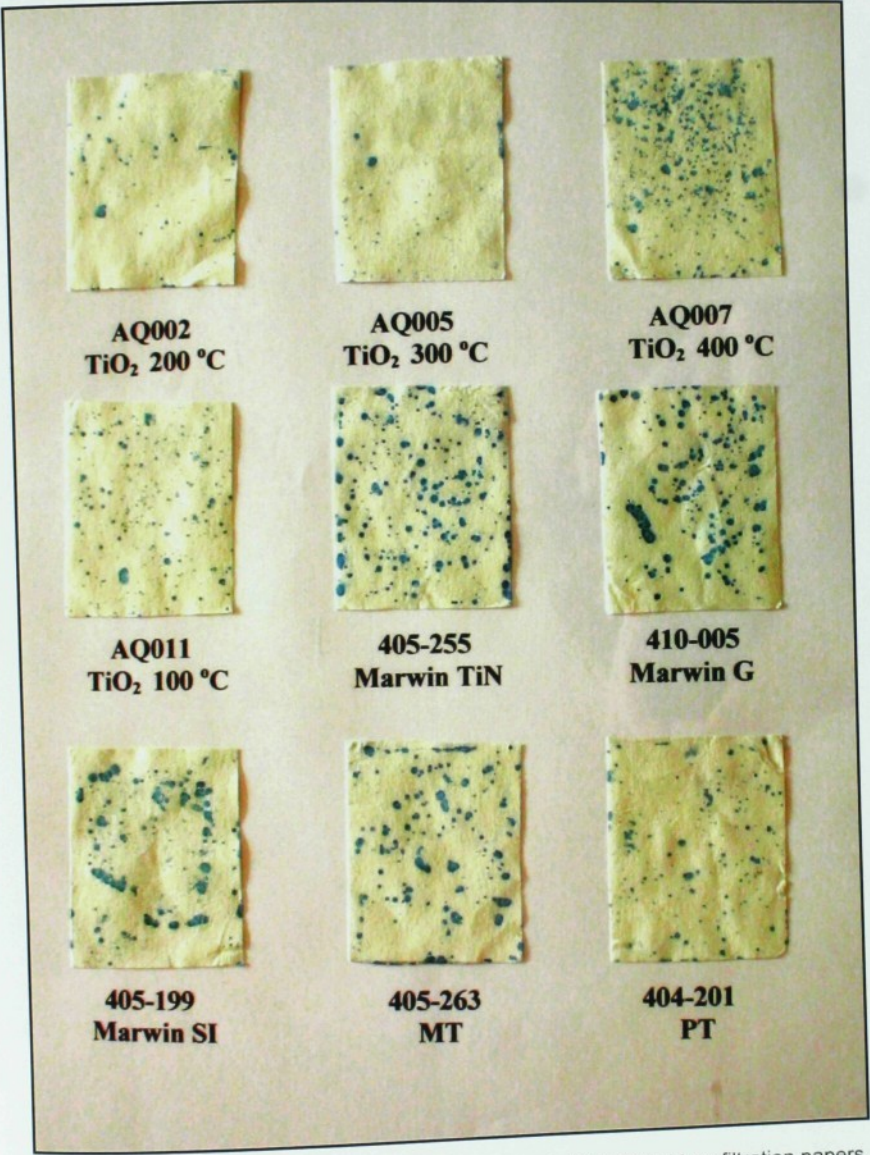


Fig 7: The results of porosity test after Czech standard ČSN 038154 on filtration papers



Fig 8: Opened humidity chamber



Fig 9: Closed humidity chamber with samples inside



Fig 10: Stand with all panels used in corrosion test cycle 4



Fig 11: Stand with all panels used in corrosion test cycle 4

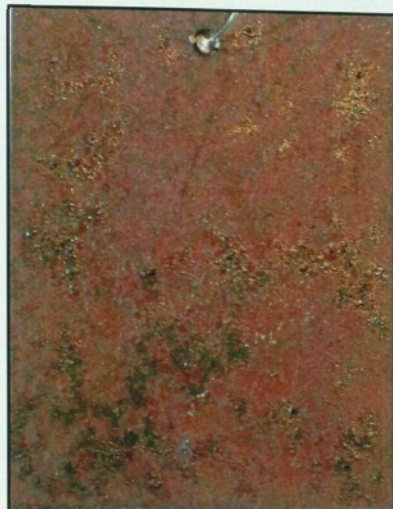


Fig 12: Panel of TiOx 100°C coating after corrosion test cycle 4



Fig 13: Panel of TiOx 200°C coating after corrosion test cycle 4



Fig 14: Panel of TiOx 300°C coating after corrosion test cycle 4



Fig 15: Panel of TiOx 400°C coating after corrosion test cycle 4



Fig 16: Panel of Marwin TiN coating after corrosion test cycle 4



Fig 17: Panel of Marwin SI coating after corrosion test cycle 4



Fig 18: Panel of MT coating after corrosion test cycle 4



Fig 19: Panel of PT coating after corrosion test cycle 4



Fig 20: Panel of Marwin-G coating after corrosion test cycle 4



Fig 21: Panel A of polyethylene coating after corrosion test cycle 4



Fig 22: Panel B of polyethylene coating after corrosion test cycle 4



Fig 23: Panel D of polyethylene coating after corrosion test cycle 4



Fig 24: Panel of Progakor Metal E coating after corrosion test cycle 4



Fig 25: Panel of uncoated thick substrate after corrosion test cycle 4



Fig 26: Panel of uncoated thin substrate after corrosion test cycle 4



Fig 27: Panel of uncoated thin substrate after corrosion test cycle 4



Fig 28: Panel of uncoated 13.155g substrate after corrosion test cycle 4



Fig 29: Panel of TiOx100°C coating after corrosion test cycle 9



Fig 30: Panel of TiOx200°C coating after corrosion test cycle 9



Fig 31: Panel of TiOx300°C coating after corrosion test cycle 9



Fig 32: Panel of TiOx400°C coating after corrosion test cycle 9



Fig 33: Panel of Marwin-G coating after corrosion test cycle 9



Fig 34: Panel of Marwin-TiN coating after corrosion test cycle 9



Fig 35: Panel of Marwin-Si coating after corrosion test cycle 9



Fig 36: Panel of MT coating after corrosion test cycle 9



Fig 37: Panel of PT coating after corrosion test cycle 9



Fig 38: Panel D of polyethylene coating after corrosion test cycle 9



Fig 39: Panel B of delaminated PE coating after corrosion test cycle 9



Fig 40: Panel A of delaminated PE coating after corrosion test cycle 9



Fig 41: Panel of uncoated 13,155g after corrosion test cycle 9



Fig 42: Panel of uncoated thin after corrosion test cycle 9



Fig 43: Panel of uncoated thick after corrosion test cycle 9



Fig 44: Panel of pragokor Metal E paint coating after corrosion test cycle 9



Fig 45: Panel PT after corrosion removed

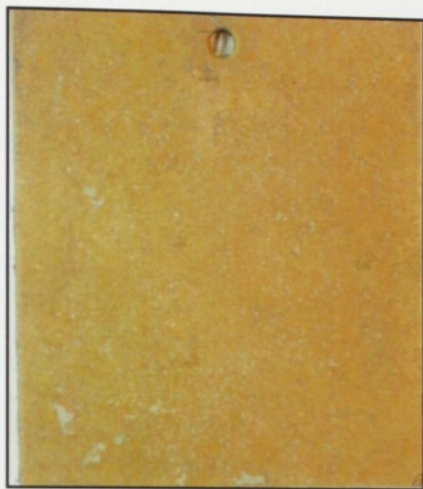


Fig 46: Panel MT after corrosion removed



Fig 47:Panel Marwin-SI after corrosion removed

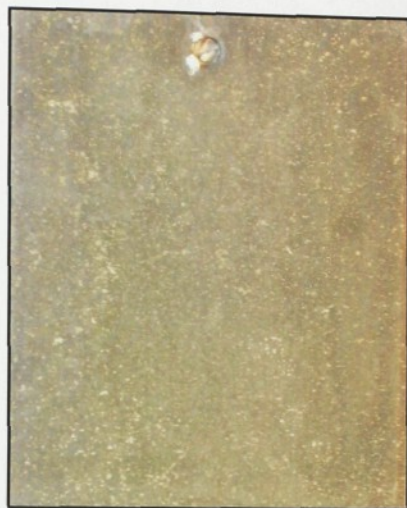


Fig 48:Panel Marwin-G after corrosion removed



Fig 49:Panel Marwin-TiN after corrosion removed



Fig 50: Panel TiOx 100°C after corrosion removed



Fig 51: Panel TiOx 200°C after corrosion removed



Fig 52 :Panel TiOx 300°C after corrosion removed



Fig 53: Panel TiOx 400°C after corrosion removed



Fig 54: Panel D polyethylene after corrosion removed

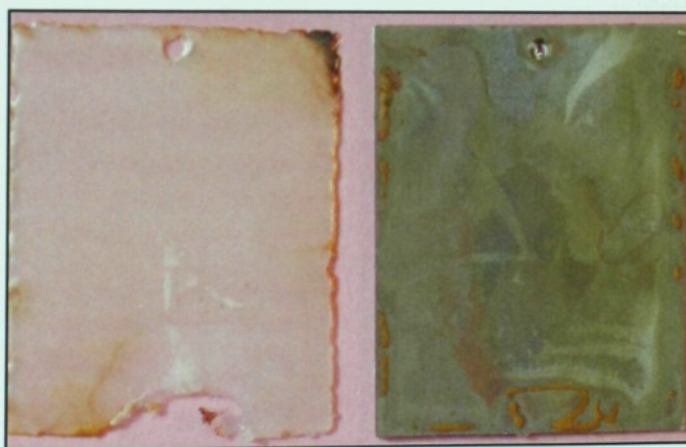


Fig 55: Panel B polyethylene after corrosion removed

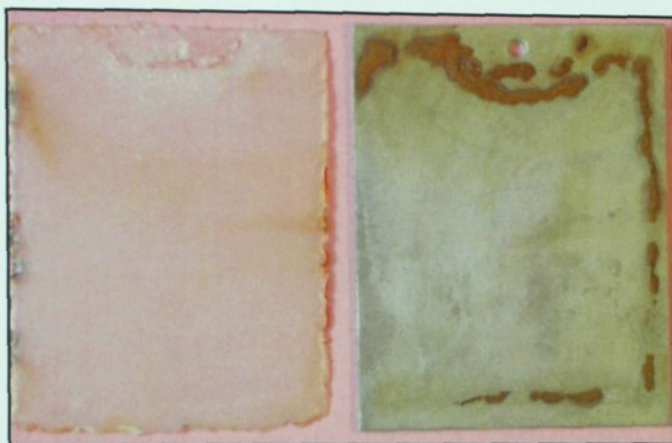


Fig 56:Panel A polyethylene after corrosion removed