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SUMMARY
DOCTORAL THESIS

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Bacău, 2026

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THEORETICAL AND EXPERIMENTAL STUDIES REGARDING THE ACTION OF CORROSIVE MEDIA ON METALLIC MATERIALS USED IN INDUSTRY

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CHAPTER 1. ANALYSIS OF THE CURRENT STATE OF RESEARCH REGARDING THE CORROSIVE ACTION OF MICROBIOLOGICAL AGENTS ON METALLIC MATERIALS

Corrosion represents the process of destruction of metals and alloys as a result of chemical, electrochemical, and biochemical interactions with their surrounding environment. This process is undesirable in industry because it causes major damages. Corrosion may be classified according to the mechanism by which it develops and according to the appearance of the damage, as follows: chemical corrosion, electrochemical corrosion, atmospheric corrosion, biological corrosion, continuous corrosion, and localized corrosion.

Biological corrosion is caused by the presence of microorganisms within the acting environment. Humid environments represent favorable conditions for the development of microorganisms. The microorganisms responsible for corrosion include bacteria, fungi, and algae, which using carbon-containing molecules as an essential nutrient source can develop on any medium where such molecules are available. Accumulated biofilms can form biodeposits that may reach several centimeters in diameter. These biodeposits can significantly affect the chemical environment at the metal/film interface or at the metal/deposit interface.

Certain species present in the composition of a biofilm produce exopolymers that coat the cells and extend their surface both into the substrate and throughout the fluid. The extracellular polymer is composed of polysaccharides and water and may provide increased environmental protection for the survival of the microorganism. Polysaccharides constitute an important carbon source required for the development of microorganisms.

In general, metals react with most chemical elements present in the atmosphere. Oxygen has the most damaging effect on metals due to the formation of oxide films. These films provide protective action only at a thickness of 400–5000 Å. In the case of iron, atmospheric oxidation leads to the formation of iron oxides.

Protection against corrosion represents the totality of measures taken to shield technical materials from the action of corrosive environments. In some cases, corrosion prevention can be achieved by protecting metals with different chemical substrates existing in the environment. Corrosion protection may also be ensured through the use

of the following protective solutions: corrosion inhibitors, polymers, and cathodic or anodic protection. Classical methods of corrosion protection include cladding, electroplating, enameling, galvanizing, and electroforming. All these methods are beneficial depending on the metallic surface being treated.

CHAPTER 2. OBJECTIVES AND STAGES OF THE DOCTORAL THESIS

The present work aims primarily to analyze and reduce the effects of microbiological corrosive agents on various metallic materials. The analysis of the effects of microbiological agents on metallic materials will allow the introduction or elimination of certain essential operations even from the design phase of metallic components. Reducing the effects of microbiological agents will enable the establishment of an environmental framework favorable to optimizing mechanical properties by inhibiting the rapid development of microorganisms present in the environment.

To achieve the proposed objectives, the thesis will approach the following research directions:

- Identification through theoretical research of the products resulting from the biocorrosion mechanism;
- Verification of the effects of microbiological corrosive agents using the following techniques: determination of mechanical properties through tensile–compression testing, analysis of deformability characteristics using the Marciniak method, analysis of stress states induced in metallic materials by the corrosive action of microorganisms using the hole-drilling method, study of the surface quality of metallic materials resulting from the action of microbiological agents using an atomic force microscope.
- Application of a 3^3 factorial experimental program to determine a medium capable of inhibiting the multiplication of microorganisms present in the environment that exert corrosive action on metallic materials.

CHAPTER 3. THEORETICAL CONTRIBUTIONS REGARDING THE BIOCORROSION PROCESS GENERATED BY THE ACTION OF MICROORGANISMS PRESENT IN DIFFERENT CORROSIVE ENVIRONMENTS ON METALLIC MATERIALS

All cells, whether bacterial or of another nature, require various nutrients, the most important of which are sources of carbon and energy. Based on these elements, the cells carry out a series of coordinated and regulated biochemical reactions known as metabolic activities.

Oxidation–reduction reactions create a flux of chemical compounds through the cells of microorganisms. Electron donors or acceptors required for these redox reactions are taken from the surrounding environment.

The acceleration of the electrochemical corrosion phenomenon is caused by the metabolic activity of microorganisms through two major effects. The first consists of the use of sulfate as an electron acceptor, and the second consists of the use of hydrogen as an electron donor.

In the case of bacterial corrosion, the responsibility for localized corrosion in steels should not be attributed directly to the observed acidity, since many microorganisms are capable of acidifying the environment.

Aluminum alloys exhibit high mechanical strength associated with good plasticity, being able to deform plastically at cold temperatures very easily, and they also possess high corrosion resistance and good weldability. Corrosion resistance is achieved through the formation of the chemical compound $MnAl_6$ and the incorporation of Fe ions into the alloy, resulting in the compound $(MnFe)Al_6$.

The effect of bacteria on steels and aluminum alloys depends on physicochemical conditions: aerobiosis (oxygenated environments), strict anaerobiosis (deoxygenated environments), or mixed conditions (oxygenated and deoxygenated). Oxygen penetrates very poorly between the area beneath the deposit (which becomes oxygen-depleted and thus anodic) and the outer area of the deposit. The outer area is oxygenated and becomes the cathode of the electrochemical cell formed between the two zones. In the absence of oxygen, the corrosion rate of steel and aluminum alloy is low due to the non-acidic nature of the liquid environment.

The mechanisms proposed for aerated and de-aerated conditions apply only to the regions corresponding to the biofilm. To these mechanisms, the conditions of differential aeration are added, which lead to an unfavorable synergy for the material, where the most severe degradations are observed.

The effect of microorganisms on the behavior of steels and aluminum alloys is related to the chemical changes (pH and the presence of sulfur compounds) imposed by the surrounding environment.

According to the electrochemical hypothesis, the destruction of metals in corrosive environments occurs because the metallic structure continuously contains free anodic paths, enabling selective dissolution through the electrochemical mechanism.

CHAPTER 4. EXPERIMENTAL RESEARCH REGARDING THE EFFECT OF MICROBIOLOGICAL CORROSIVE AGENTS ON THE MECHANICAL CHARACTERISTICS OF METALLIC MATERIALS

After 30 days during which the corrosive environments acted upon the metallic specimens, the samples were removed from their respective media and subjected to mechanical testing aimed at determining the mechanical properties specific to each specimen following exposure to the corrosive conditions.

Results of the experimental tests for the material steel

The mechanical properties of the metallic materials determined through experimental testing for the different corrosive environments are presented in Table 1.

Table 1. Mechanical properties of steel

Metallic material	*Aggressive corrosive environment*		*Young's modulus,[MPa]*	*Yield strength, [MPa]*	*Total elongation,%*	*Anisotropy coefficient, r*	*Strain hardening coefficient, n*
Steel	*Natural*		*203275*	*354,555*	*22,7*	*1,86135*	*0,1713*
	Saline	*Control*	*942850*	*343,72*	*16*	*1,70558*	*0,2019*
		7%	*101466,5*	*343,775*	*16*	*1,58601*	*0,1538*
		11%	*99112*	*345,32*	*21*	*1,65319*	*0,1601*
	Microbiological	*Control*	*94841*	*349,59*	*14*	*1,18605*	*0,1673*
		Fungus	*101165*	*329,485*	*12*	*1,45378*	*0,1739*
		Mould	*107510*	*342,64*	*12*	*1,69717*	*0,1697*

The steel reference specimen represents the sample on which the microorganisms naturally present on its surface acted, namely: bacteria, filamentous fungi, and molds.

From the analysis of the stress–strain diagrams for the microbiological environment, slightly different curve levels can be observed compared to those obtained in the ambient environment; however, the action of the microorganisms resulted in a narrower yield region and a more rapid fracture of the specimens.

Results of the experimental tests for the material: aluminum alloy

The mechanical properties obtained after subjecting the metallic material, aluminum alloy, to the action of the corrosive environments are presented in Table 2.

Tabelul 2. Mechanical properties of aluminium alloy

Metallic material	*Aggressive corrosive environment*		*Young's modulus,[MPa]*	*Yield strength, [MPa]*	*Total elongation,%*	*Anisotropy coefficient, r*	*Strain hardening coefficient, n*
Aluminium alloy	*Natural*		*72624,5*	*292,7*	*17,95*	*3,8016*	*0,18860679*
	Saline	*Control*	*75594*	*290,87*	*21,05*	*2,8783*	*0,20353545*
		7%	*52646*	*294,74*	*10,172*	*4,2005*	*0,23156841*
		11%	*45784*	*290,84*	*9,767*	*2,6702*	*0,22568784*
	Microbiological	*Control*	*47138*	*298,14*	*12*	*4,0589*	*0,23790962*
		Fungus	*45969*	*295,48*	*11,775*	*3,9799*	*0,24160467*
		Mould	*45336*	*295,31*	*11,815*	*5,4799*	*0,23629911*

From the analysis of the graphs, a significantly higher level of the stress–strain curves can be observed for the specimen subjected to the action of the saline environment – reference sample – compared to the ambient environment, as well as a narrowing of the yield region and a faster fracture in the case of the 7% and 11% saline environments.

The action of the microbiological environment on the aluminum alloy diminishes its corrosion resistance and, implicitly, its strength characteristics. The modification of the mechanical properties of the material occurs due to the formation of corrosion products resulting from the activity of various microorganisms developed on the surface of the specimen.

Results of the experimental tests for the ambient corrosive environment

The stress–strain curve for the aluminum alloy shows a lower level compared to the curves obtained for the steels. The differences in the levels of the stress–strain curves for the steels are due to the differences in their chemical compositions. For example, metals belonging to secondary subgroups, where Fe is also included, tend to

passivate, a fact that explains the increase in their corrosion resistance and the differences between their mechanical characteristics.

Results of the experimental tests for the corrosive environment: saline-reference

The stress-strain curve for the aluminum alloy shows a lower level compared to the curves obtained for the steels. Thus, distilled water provides the aluminum alloy and steel specimens with high corrosion resistance, resistance attributed to the high magnesium and carbon content in these alloys. The narrower yield regions observed in the stress-strain curves for the aluminum alloy and steel indicate lower plasticity, resulting in faster fracture.

Results of the experimental tests for the corrosive environment: saline 7%

From the analysis, it can be observed that the 7% saline environment induces narrower yield regions and faster fracture in both the aluminum alloy and steel. This behavior is influenced by the presence of Na^+ and Cl^- ions, which form networks with the metallic ions at the surface of both steel and the aluminum alloy. Furthermore, the formation of metallic oxides leads to an additional narrowing of the yield region of the materials.

Results of the experimental tests for the corrosive environment: saline 11%

In the 11% saline corrosive environment, similarly to the 7% saline environment, both the aluminum alloy and steel exhibit higher stress-strain curve levels, narrower yield regions, and faster fracture.

Results of the experimental tests for the corrosive environment: microbiological – reference

In the microbiological reference environment, several species of microorganisms are present and act upon the metallic materials, influencing their behavior under mechanical loads. Thus, the variation curves for steel and the aluminum alloy indicate a lower yield domain and faster fracture compared to the other

environments. These characteristics are influenced by the development of microorganisms on the material surface, microorganisms that obtain the necessary metabolic energy from the oxidation of the metal at the surface.
In the case of these samples, microorganisms affect the material surface in two ways: directly, by developing an oxidation–reduction equilibrium or an electrochemical system, or indirectly, through the formation of nodules or ferric hydroxide precipitates.

Results of the experimental tests for the corrosive environment: microbiological

The levels and shapes of the stress–strain curves for steel and the aluminum alloy show narrower yield regions and faster fracture due to the pronounced action of the corrosive environment. This effect is explained by the fact that the metabolism of microscopic fungi produces a series of organic acids that solubilize metals and reduce their resistance to mechanical loading. The corrosive action of fungi on metals is more pronounced in warm and humid areas. The effect of microscopic fungi on these materials is explained by their heterotrophic nutrition mode, their enzymatic properties, and the formation of organic acids (gallic, citric, oxalic, etc.).

Results of the experimental tests for the corrosive environment: microbiological – mold

For both steel and the aluminum alloy, narrower yield regions and faster fracture are observed-characteristics influenced by the presence of molds, which can attach much more rapidly to steel and aluminum, diminishing their corrosion resistance.

ANALYSIS OF THE EFFECTS OF CORROSION ON THE DEFORMABILITY OF METALLIC MATERIALS

For this purpose, circular specimens made of homogeneous materials (steel) were utilized. Prior to testing, the specimens were either exposed or not exposed to the corrosive action of the microorganism-containing corrosive environment. The dimensions of the specimens used are presented in Table 3.

Table 3. Dimensional characteristics of the specimens for the Marciniak test

Samble	*1*	*2*	*3*	*4*	*5*
Width (A) [mm]	*50*	*90*	*110*	*140*	*200*
Radius (R) [mm]	*110*	*95*	*80*	*65*	*0*

Experimental Test Results

The plotting of the forming limit curves was performed using the 7D software, which provides the exact position of the minimum and maximum logarithmic strains.

Based on the analysis of the forming limit curves obtained for steel, the following aspects can be observed:

- ✓ The forming limit curves exhibit different levels for corroded steel compared with uncorroded steel.
- ✓ The curve corresponding to uncorroded steel is positioned at a higher level, indicating a higher degree of deformability of the metallic material during plastic deformation.
- ✓ The curve corresponding to corroded steel lies at a lower level, as the deformability is negatively influenced by the metabolic products of the microorganisms, which adversely affect both the material quality and its resistance to deformation.

From the analysis of the curves obtained for steel, the overall levels nevertheless indicate a better deformability and a corrosion resistance associated with the products formed during the biocorrosion mechanism.

CONCLUSIONS REGARDING THE EXPERIMENTAL ANALYSIS OF THE EFFECT OF BIOCORROSION ON THE MECHANICAL PROPERTIES OF METALLIC MATERIALS

In the saline control environment, distilled water-having no pronounced corrosive action-does not significantly contribute to the alteration of the mechanical properties of metallic materials. In contrast, saline environments with concentrations of 7% or 11% induce a lower plasticity in steel and aluminum alloy materials. The behavior of materials in the saline environment is determined by the presence of Na^+ and Cl^- ions, which may more easily or more difficultly form networks with the metallic ions on the surface of the materials, as well as by the phenomenon of deposition or non-deposition of limestone and carbonates, which can isolate the metal from water and thus slow down the corrosion process.

In the microbiological environment, where several species of microorganisms are present, these organisms act upon the metallic materials, influencing their behavior under mechanical loading. The development of microorganisms that obtain the energy required for their metabolism through the oxidation of the surface metal is more pronounced in steel and aluminum alloy materials. Furthermore, the metabolic activity of microscopic fungi produces various organic acids, which lead to the solubilization of these metals. For these reasons, the corrosion resistance of such materials decreases, resulting implicitly in a reduction of their plasticity.

The chemical composition of the materials influences their deformability characteristics. Steel, due to its chemical composition, specifically its high Mn and C content, exhibits lower deformability capacities. Purity positively influences the deformability of the material by ensuring homogeneous deformation, whereas impurities promote crack initiation at their interface with the metallic matrix.

The attack of microbiological agents on the quality of metallic materials (as in the case of corroded steel) and the consequent reduction in their deformation capacity, as evidenced by the lower level of the forming limit curves, can be explained by the fact that the metabolism of the fungi and molds developing on the material generates a series of organic acids (most commonly tartaric, citric, and oxalic acids). These acids solubilize the metals and form microbial cells with differentiated concentration, which in turn affect the structure of the materials.

CHAPTER 5. EXPERIMENTAL INVESTIGATIONS REGARDING THE EFFECT OF VARIOUS CORROSIVE AGENTS ON THE SURFACE LAYERS OF METALLIC MATERIALS

Surface Analysis of the Metallic Material Steel

The 3D projection of steel in the ambient environment is presented in Figure 1, while its comparative projection in the 7% saline environment is shown in Figure 2. The projections correspond to a scanned surface area of 100 μm^2, on which corrosion can be observed to a depth of 1.9 μm, caused by the presence of NaCl in the environment.

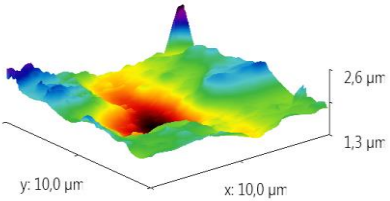


Figure 1. 3D projection of metallic material steel under ambient conditions

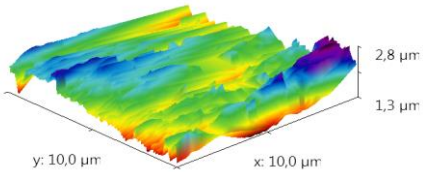


Figure 2. 3D projection of metallic material steel under the action of a 7% saline environment

In Figure 3, the texture and roughness of the material under the action of the ambient environment are presented, while in Figure 4 this texture is shown in comparison with the roughness of the material subjected to the 7% saline environment.

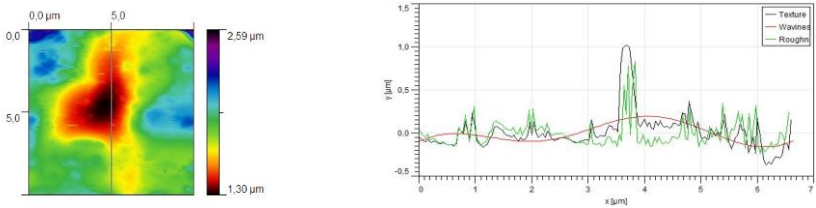


Figure 3. Acquired image and topography of metallic material steel under ambient conditions

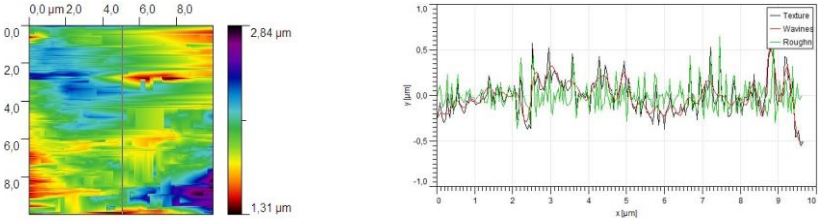


Figure 4. Captured image and surface topography of metallic material steel exposed to a 7% saline environment

From the analysis of the two textures, a denser and finer roughness can be observed in the case of the 7% saline environment, compared with the ambient environment.

Figure 5 presents the 3D projection of the metallic material steel under the action of the 11% saline environment, in comparison with the ambient environment. The 3D projections highlight the depth of the corrosive action, which reaches 1.2 µm.

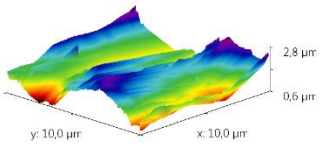


Figure 5. 3D representation of metallic material steel exposed to an 11% saline environment

The texture and roughness of the material after exposure to the 11% saline environment are presented in Figure 6. From the texture analysis, larger-scale surface irregularities can be observed compared with those produced by the 7% saline environment; however, their number is significantly lower due to the deposition of limestone from the more concentrated NaCl solution.

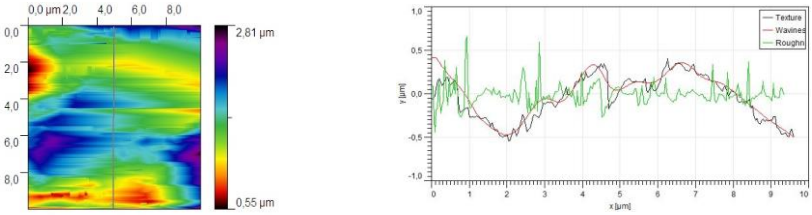


Figure 6. Captured image and surface topography of metallic material steel 1 exposed to an 11% saline environment

The 3D projections in the microbiological control environment and in the microbiological-fungal environment are presented in Figures 7 and 8.

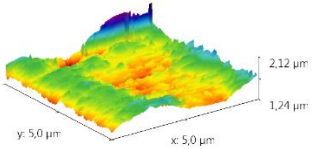


Figure 7. 3D representation of metallic material steel exposed to the microbiological environment – control sample

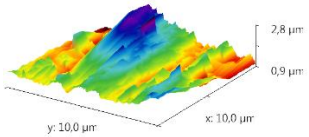


Figure 8. 3D representation of metallic material steel exposed to the microbiological environment – fungus

From the analysis of the projections, it can be observed that the action of the microbiological, fungal environment is similar to that of the 7% saline environment, with dense surface asperities of small magnitude being present.

Figures 9 and 10 present the textures and surface roughness of the material under the action of the two microbiological environments. The 3D projection in the microbiological–mold environment is shown in Figure 11.

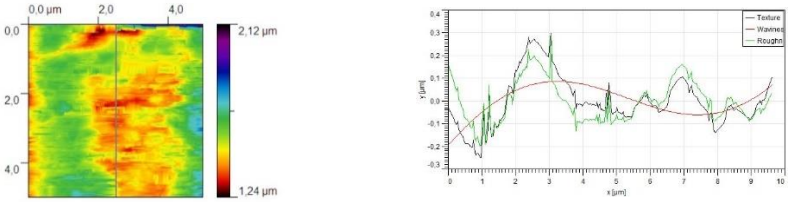


Figure 9. Captured image and surface topography of metallic material steel exposed to the microbiological environment – control sample

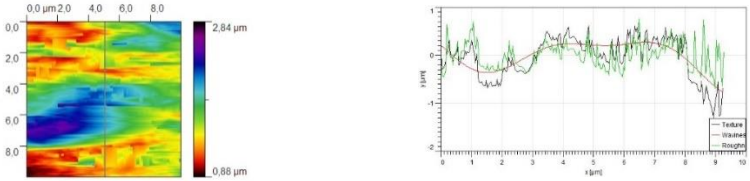


Figure 10. Captured image and surface topography of metallic material steel exposed to the microbiological environment

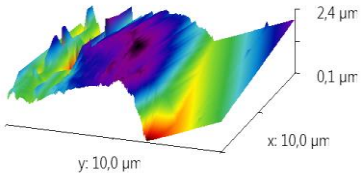


Figure 11. 3D representation of metallic material steel 1 exposed to the microbiological environment – mold

From the analysis of the projections, it can be observed that the action of the mold on the metallic material manifests through the formation of sparse yet deep surface asperities.

Figure 12 presents the surface topography of the material under the action of the microbiological-mold environment.

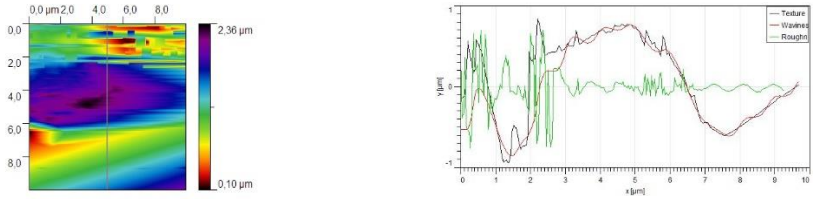


Figure 12. Captured image and surface topography of metallic material steel exposed to the microbiological environment – mold

Surface Analysis of the Aluminum Alloy Metallic Material

The 3D projection of the surface of the aluminum alloy material in the ambient environment is presented in Figure 13. Figure 14 shows the 3D projection in the 7% saline environment, which, compared with the ambient environment, exhibits corrosion to a depth of 1.9 µm on a scanned surface area of 10 µm².

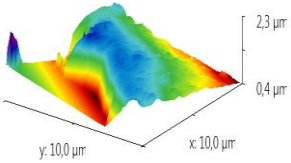


Figure 13. 3D representation of the Al alloy metallic material exposed to ambient conditions

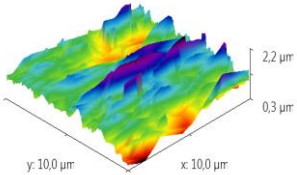


Figure 14. 3D representation of the Al alloy metallic material exposed to a 7% saline environment

The acquired images and the surface texture under the action of the two corrosive environments-ambient and 7% saline are presented in Figures 15 and 16.

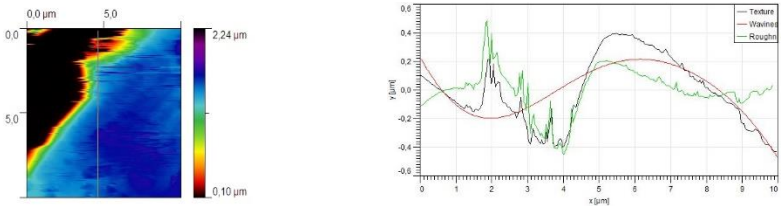


Figure 15. Captured image and surface topography of the Al alloy metallic material exposed to ambient conditions

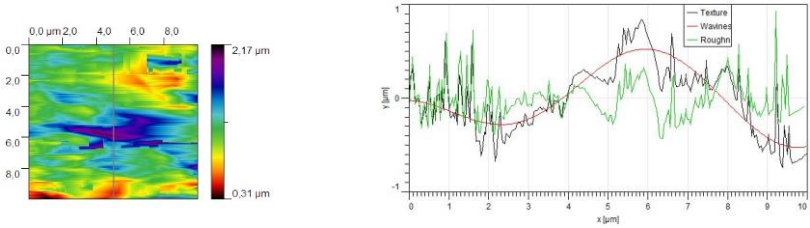


Figure 16. Captured image and surface topography of the Al alloy metallic material exposed to a 7% saline environment

Compared with the ambient environment, no significant difference in depth corrosion is observed; the corrosion occurs mainly at the material interface. Thus, the 7% concentration does not substantially influence the corrosion of the material over a period of 30 days.

The projection in the 11% saline environment, compared with the ambient environment, is presented in Figure 17. From the analysis of the projection on a scanned surface area of 10 µm², corrosion to a depth of 2.4 µm can be observed.

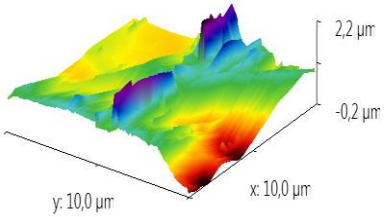


Figure 17. 3D representation of the Al alloy metallic material exposed to an 11% saline environment

The acquired image and the surface texture under the action of the 11% saline corrosive environment are presented in Figure 18. In this case, a deeper corrosion process can be observed; however, a smaller amount of NaCl is deposited on the surface, limiting its action on the aluminum alloy material.

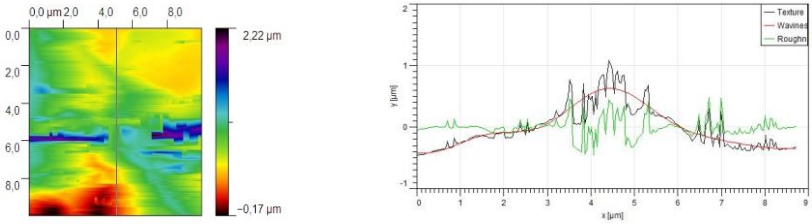


Figure 18. Captured image and surface topography of the Al alloy metallic material exposed to an 11% saline environment

The analysis of the surface texture of the aluminum alloy metallic materials under the action of the 11% saline environment highlights the transformation of the initially smooth surface into one characterized by asperities.

The 3D projection in the microbiological control environment is presented in Figure 19, while the 3D projection in the microbiological–fungal environment is shown in Figure 20. In the microbiological control environment, where microorganisms developed on the nutrient medium acted upon the material, a smooth surface is observed. In contrast, the fungi acted on the material by inducing asperities penetrating up to 0.64 µm into the material.

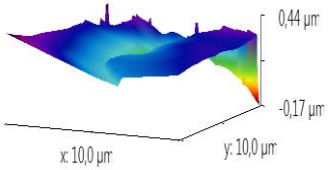


Figure 19. 3D representation of the Al alloy metallic material exposed to the microbiological environment – control sample

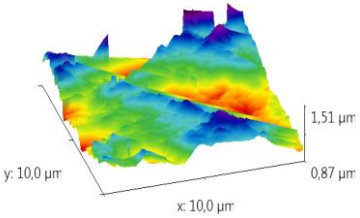


Figure 20. 3D representation of the Al alloy metallic material exposed to the microbiological environment – fungi

In Figure 21, the acquired image and the texture of the aluminum alloy material under the action of the microbiological control environment are presented, while Figure 22 shows the acquired image and the surface texture of the material under the action of the microbiological environment. The texture of the material is influenced by the action of the fungi or molds, although a certain resistance and rigidity of the material are still maintained during this interaction.

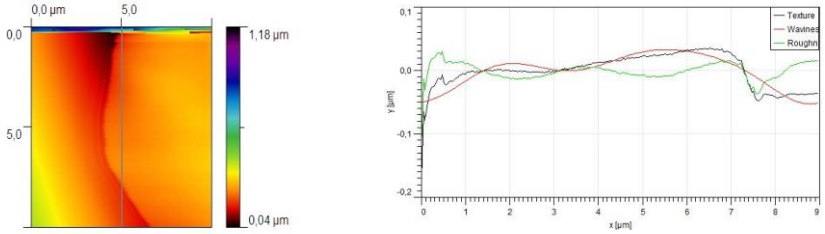


Figure 21. Captured image and surface topography of the Al alloy metallic material exposed to the microbiological environment – control sample

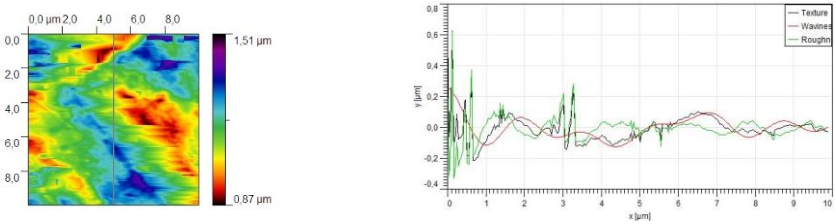


Figure 22. Captured image and surface topography of the Al alloy metallic material exposed to the microbiological environment – fungus

Figure 23 presents the 3D projection corresponding to the microbiological–mold environment. Compared with the action of the ambient environment, a significant difference can be observed between the two projections, with the microbiological–mold environment producing dense surface asperities of small dimensions.

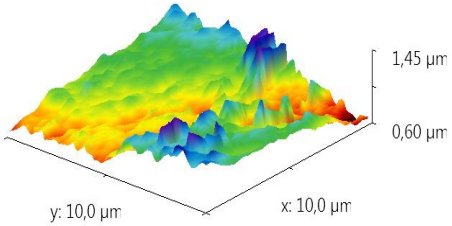


Figure 23. 3D representation of the Al alloy metallic material exposed to the microbiological environment – mold

In Figure 24, the image and the surface texture of the material under the action of the mold–fungal environment are presented. From the analysis of the images, a

corrosive action of this environment on the material can be observed, reaching a depth of 0.85 µm within the material.

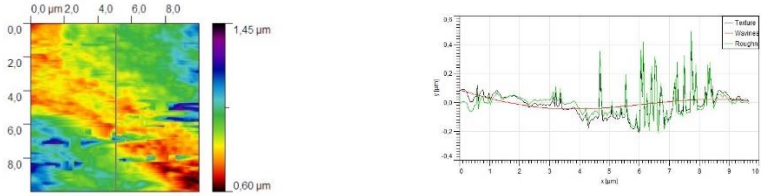


Figure 24. Captured image and surface texture of the Al alloy metallic material exposed to the microbiological environment – mold

ANALYSIS OF THE EFFECTS OF CORROSION ON THE DISTRIBUTION OF RESIDUAL STRESSES IN METALLIC MATERIALS

The analysis of the curves for steel, in both corroded and uncorroded states, shows a small difference regarding the curve levels and the localization of the stress values across the thickness of the material. In the case of the specimens subjected to corrosion, a slight increase in the depth at which higher stress values occur can be observed. This indicates that the corroded layer affects both the distribution and the magnitude of the residual stresses. Furthermore, considering the thickness of the material, a difference can be noticed between the stress distributions in the regions near the two faces of the metallic material.

In the case of the aluminum alloy material, whether corroded or uncorroded, a more pronounced difference in residual stress values is observed between the two conditions, namely, lower stress values in the uncorroded specimen and higher stress values in the corroded specimen. Therefore, the chemical composition of the aluminum alloy favors the development of microorganisms, making the corroded material more susceptible to premature failure compared with the uncorroded material. Additionally, for the specimens subjected to corrosion, a difference is observed between the stress distributions near the two surfaces of the material, as well as an increase in the depth at which higher stress values occur. Thus, similar to the case of steel, the corroded layer affects both the distribution and the magnitude of the residual stresses.

CONCLUSIONS REGARDING THE ANALYSIS OF THE EFFECTS OF CORROSION ON SURFACE LAYERS

Conclusions on the Effect of Corrosion on Surface Texture and Roughness

For the steel metallic material, the saline environment influences the specimen proportionally to the degree of salinity. Thus, the 7% saline environment produces a dense and fine roughness on the specimen's surface, whereas the 11% saline environment generates surface asperities of significantly greater magnitude than those in the 7% saline medium, although fewer in number.

For the aluminum alloy material, the saline environment affects the surface by transforming it from a relatively smooth, asperity-free surface into a rough one; based on the 3D images, the salinity level does not significantly influence the degree of surface roughness. From a microbiological perspective, only the mold-contaminated microbiological environment has a notable effect on the specimen, modifying its surface through the appearance of dense, small-scale asperities.

Conclusions on the Effect of Corrosion on Stress Distribution Across the Thickness of Metallic Materials

The metallic materials selected for analysis exhibit significant differences in the levels of the variation curves and in the residual stress values between corroded specimens and those not subjected to the corrosive action of microorganisms. In both materials, due to the effects of microbiological agents, higher residual stress values are induced compared with the uncorroded condition.

During the 30-day exposure period to corrosive agents, microbiological agents acted not only on the surface but also penetrated into the depth of the materials, modifying the residual stress state across the thickness. This may lead to the initiation and rapid propagation of cracks, particularly because the stresses generated are exclusively tensile stresses.

Differences also arise between the stress distributions in the regions near the two surfaces of the metallic material, which may significantly influence changes in the shape and dimensions of metallic components through the phenomenon of elastic springback.

CHAPTER 6. CONTRIBUTIONS TO REDUCING THE CORROSIVE ACTION OF MICROBIOLOGICAL AGENTS ON METALLIC MATERIALS THROUGH THE APPLICATION OF FACTORIAL MATHEMATICAL MODELS

For the purpose of this study, a 3^3-type factorial experiment was designed, which required the execution of 27 experimental tests. The metallic materials used were the same as in the previous studies, being exposed to the action of microorganisms. These microorganisms developed on a solid Malt-Agar medium with variable pH, under controlled modifications of temperature and development duration.

Based on the calculations performed using the derivative method, the optimal values of the parameters that lead to favorable mechanical characteristics for the studied steels were established. This stage was influenced by the following three variables: pH (x_1); the development temperature of the microorganisms (x_2); and the duration of microorganism action (x_3). Three levels of variation were considered for each variable, as presented in Table 4.

Table 4. Considered parameters and their variation ranges

Parameter	*Reduced variable*	*Base values (0)*	*Minimum values (-1)*	*Maximum values (+1)*	*Step*
pH	X_1	*7*	*5*	*9*	*2*
Temperature	X_2	*30*	*20*	*40*	*10*
Time	X_3	*480*	*240*	*720*	*240*

The response functions considered in this study were the yield strength, total elongation, and the anisotropy coefficient of the metallic sheets analyzed. Table 5 presents the values of the yield strength, total elongation, and anisotropy coefficient as a function of the variation of the parameters, with the reduced values of the variables indicated in parentheses.

Table 5. Variable values and response functions

No.	*pH*	*Temperature,°C*	*Time, h*	*Yield strength [MPa]*	*Total elongation [%]*	*Anisotropy coefficient*
	x_1	x_2	x_3	Y_1	Y_2	Y_3
1	*(-1) 5*	*(-1)20*	*(-1)240*	*288,78*	*22,7*	*1,861*
2			*(0)480*	*270,95*	*21*	*1,705*
3			*(+1)720*	*269,45*	*18*	*1,585*
4		*(0)30*	*(-1)240*	*302,58*	*24*	*1,653*
5			*(0)480*	*289,55*	*21,05*	*1,186*
6			*(+1)720*	*302,15*	*25*	*2,453*
7		*(+1)40*	*(-1)240*	*345,78*	*37*	*1,697*
8			*(0)480*	*334,15*	*35*	*2,584*
9			*(+1)720*	*330,99*	*28,5*	*1,987*

10	(0) 7	(-1)20	(-1)240	344,68	43	2,107
11			(0)480	299,99	28	2,265
12			(+1)720	288,23	27	2,638
13		(0)30	(-1)240	297,10	17,9	2,076
14			(0)480	280,15	16	3,802
15			(+1)720	269,25	13	2,878
16		(+1)40	(-1)240	270,25	11,78	4,201
17			(0)480	295,15	41	2,671
18			(+1)720	334,55	45	4,059
19	(+1) 9	(-1)20	(-1)240	298,68	47	3,979
20			(0)480	290,55	14	5,479
21			(+1)720	270,95	12	4,413
22		(0)30	(-1)240	334,15	20	2,611
23			(0)480	295,99	12	2,455
24			(+1)720	299,16	11,8	1,987
25		(+1)40	(-1)240	269,25	25	1,894
26			(0)480	269,58	10,17	2,107
27			(+1)720	252,15	9,7	3,276

To determine the significance, three tests were performed at the central point of the domain (0, 0, 0), and their results are presented in Table 6.

Table 6. Central point values of the domain

y_k^0	y_1^0	y_2^0	y_3^0
Yield strength [MPa]	*250*	*290*	*350*
Total elongation [%]	*25*	*10*	*35*
Anisotropy coefficient	*3,571*	*2,071*	*3,957*

Determination of the Significance of the Coefficients for the Response Function Y_1

The t-Student test values for each coefficient are presented in Table 7.

Table 7. Values of the Student's t-test

t_j	t_0	t_1	t_2	t_3	t_{12}	t_{13}	t_{23}	t_{11}	t_{22}	t_{33}	t_{123}
Calculated value	*30,352*	*0,883*	*0,456*	*0,771*	*2,159*	*0,389*	*1,169*	*0,252*	*0,092*	*0,665*	*0,079*

The graphs of these mathematical models, generated using the MATLAB software, are shown in Figures 25, 26, and 27.

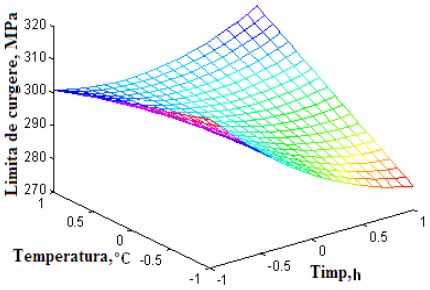


Figure 25. Influence of yield strength when the pH is maintained at its central domain level

$$Y_1 = 294.003 + 4.422 \cdot x_2 - 7.465 \cdot x_3 + 11.327 \cdot x_2 \cdot x_3 + 6.445 \cdot x_3^2$$

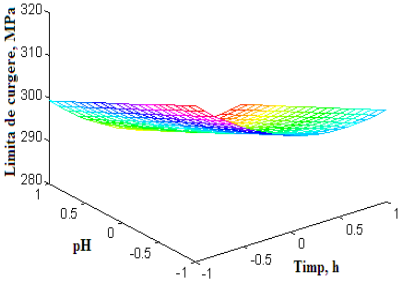


Figure 26. Influence of yield strength when the temperature is maintained at its central domain level

$$Y_1 = 294.003 - 8.551 \cdot x_1 - 7.465 \cdot x_3 + 6.445 \cdot x_3^2$$

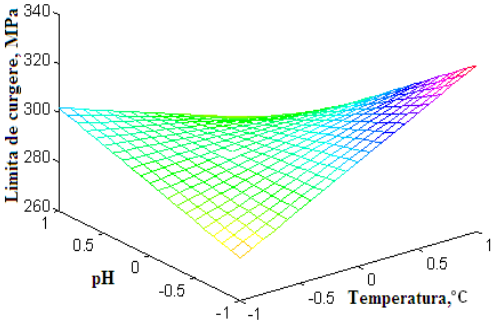


Figure 27. Influence of yield strength when the time is maintained at its central domain level

$$Y_1 = 294.003 - 8.551 \cdot x_1 + 4.422 \cdot x_2 - 20.912 \cdot x_1 \cdot x_2$$

From the graphical representations, the following aspects can be distinguished: the curvature of the surfaces, the minimum values resulting from the effects of the quadratic coefficients, and the inflection point.

Based on the actual optimum values, the following observations can be made:

- the pH value required to inhibit the rapid development of microorganisms is **8.44**;
- the microorganism development temperature at which the biocorrosion phenomenon does not occur is **26 °C**;
- the optimal exposure time of the metallic specimens to the microbiological environment is **705 h ≈ 27 days**.

From the analysis of the obtained data, it results that the yield strength of the materials subjected to the microbiological corrosive environment tends toward the optimal value when all variables remain within the limits of the initially selected variation domain.

Determination of the Significance of the Coefficients for the Response Function Y_2

The t-Student test values for each coefficient are presented in Table 8.

Tabel 8. Values of the Student's t-test

t_j	t_0	t_1	t_2	t_3	t_{12}	t_{13}	t_{23}	t_{11}	t_{22}	t_{33}	t_{123}
Calculated value	*8,433*	*1,619*	*0,24*	*1,339*	*2,303*	*1,593*	*2,241*	*2,098*	*3,541*	*0,962*	*1,213*

From the analysis of the test results, it can be observed that the following terms may be eliminated: **x_2^2, x_3^2, and $x_1x_2x_3$.**

The graphs of these mathematical models, generated using the MATLAB software, are presented in Figures 28, 29, and 30.

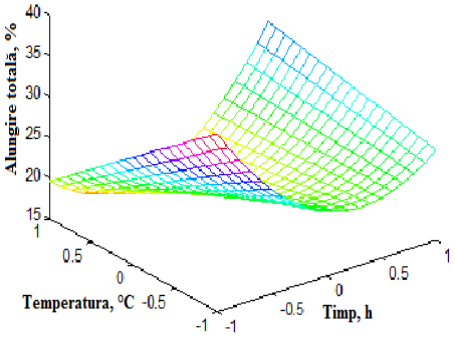


Figure 28. Influence of total elongation when the pH is maintained at its central domain level

$$Y_2 = 19.694 - 3.243 \cdot x_3 + 8.575 \cdot x_2^2 + 5.427 \cdot x_2 \cdot x_3$$

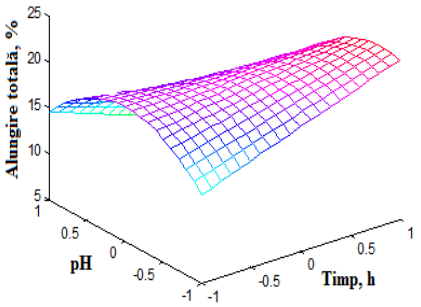


Figure 29. Influence of total elongation when the temperature is maintained at its central domain level

$$Y_2 = 19.694 - 3.921 \cdot x_1 - 5.08 \cdot x_1^2 - 3.858 \cdot x_1 \cdot x_3$$

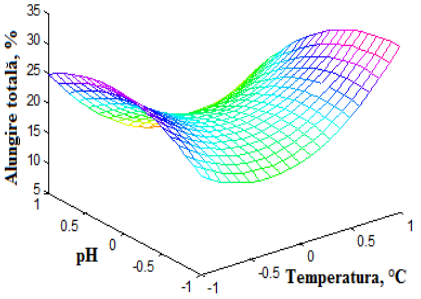


Figure 30. Influence of total elongation when the time is maintained at its central domain level

$$Y_2 = 19.694 - 3.921 \cdot x_1 - 5.08 \cdot x_1^2 + 8.575 \cdot x_2^2 - 5.577 \cdot x_1 \cdot x_2$$

From the graphical representations, the following parameters can be distinguished: the curvature of the surfaces, the minimum values resulting from the effects of the quadratic coefficients, and the inflection point. Based on the actual optimum values, the following observations can be made:

- the pH value required to inhibit the development of microorganisms is **7**;
- the microorganism development temperature at which the biocorrosion phenomenon does not occur is **36 °C**;

- the duration of action of the microbiological environment on the metallic specimens is **933.6 h ≈ 39 days**, a value that exceeds the initially selected experimental interval, thus indicating the need to reconsider the established variation domain.

The main conclusion derived from the analysis of the response function is that the total elongation of the materials exposed to the corrosive environment tends toward the optimal value when the optimal pH and temperature remain within the limits of the initially selected variation domain.

Determination of the Significance of the Coefficients for the Response Function Y_3

The t-Student test values for each coefficient are presented in Table 9.

Tabel 9. Values of the Student's t-test

t_j	t_0	t_1	t_2	t_3	t_{12}	t_{13}	t_{23}	t_{11}	t_{22}	t_{33}	t_{123}
Calculated value	*14,089*	*3,329*	*0,451*	*0,926*	*3,351*	*0,164*	*0,366*	*2,458*	*2,407*	*0,334*	*0,249*

From the t-Student test results, it can be observed that the following terms may be eliminated: **x_2x_3, x_1x_3, x_3^2, and $x_1x_2x_3$.**

Effects of the Parameters for the Response Function Y_3

The graphs of these mathematical models, generated using the MATLAB software, are presented in Figures 31, 32, and 33.

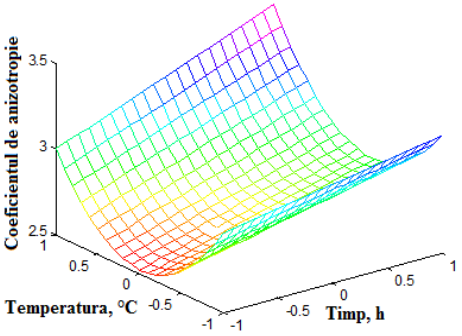


Figure 31. Influence of the anisotropy coefficient when the pH is maintained at its central domain level

$$Y_3 = 2.701 - 0.086 \cdot x_2 + 0.178 \cdot x_3 + 0.461 \cdot x_2^2 + 0.07 \cdot x_2 \cdot x_3$$

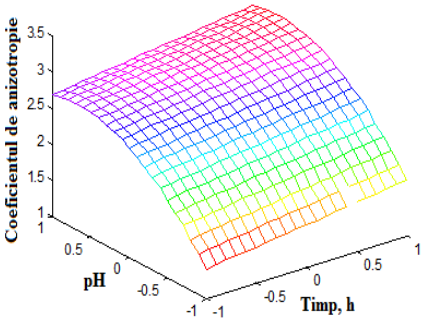


Figure 32. Influence of the anisotropy coefficient when the temperature is maintained at its central domain level

$$Y_3 = 2.701 + 0.638 \cdot x_1 + 0.178 \cdot x_3 - 0.471 \cdot x_1^2$$

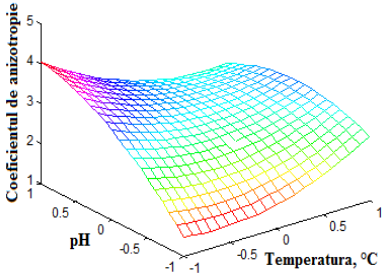


Figure 33. Influence of the anisotropy coefficient when the time is maintained at its central domain level

$$Y_3 = 2.701 + 0.638 \cdot x_1 - 0.086 \cdot x_2 - 0.471 \cdot x_1^2 + 0.461 \cdot x_2^2 - 0.643 \cdot x_1 \cdot x_2$$

From the graphical representations, the curvature of the resulting surfaces can be distinguished, and based on the actual optimum values, the following observations can be made:

- the pH value required to inhibit the development of microorganisms is **6**;
- the microorganism development temperature is **25 °C**;
- the duration of action of the microbiological environment on the metallic specimens is **907.2 h ≈ 38 days**, which exceeds the initially selected interval, indicating the need to reconsider the chosen variation domain.

The main conclusion derived from this analysis is that the response function and the anisotropy coefficient of the materials exposed to the corrosive environment tend toward the optimal value when the optimal pH and temperature remain within the limits of the initially defined variation domain.

CONCLUSIONS REGARDING THE REDUCTION OF MICROBIOLOGICAL AGENT ACTION ON METALLIC MATERIALS THROUGH THE APPLICATION OF THE 3^3 MATHEMATICAL MODEL

Following the application of the 3^3 factorial program to evaluate the action of microorganisms on steel metallic materials, the following conclusions can be drawn:

- The selected pH range of 5-9 is favorable for obtaining optimal values of the mechanical properties, yield strength, total elongation, and anisotropy coefficient. Thus, microorganisms developing on a solid Malt-Agar medium with pH values of 6, 7, and 8.44 do not significantly affect the quality of the metallic materials.
- The temperatures determined through the factorial program—25 °C, 26 °C, and 36 °C,promote the achievement of effective mechanical properties (such as yield strength, total elongation, and anisotropy coefficient) after exposure to microbiological agents.
- The exposure time plays a significant role in the quality of metallic materials subjected to the microbiological environment. Durations of 38 and 39 days of microorganism activity have an unfavorable effect on the mechanical properties of the materials studied. Therefore, based on the results obtained from the mathematical model, it has been demonstrated that a redefinition of the initial variation domain is necessary for the response functions related to total elongation and the anisotropy coefficient.

CHAPTER 7. GENERAL CONCLUSIONS. ORIGINAL CONTRIBUTIONS. FUTURE RESEARCH DIRECTIONS

GENERAL CONCLUSIONS

Corrosion is defined as the process of degradation of metals and alloys resulting from chemical, electrochemical, and biochemical interactions with their service environment.

Depending on the degradation mechanism and the nature of the damage, corrosion can be classified as follows:

- Chemical corrosion – degradation of metals and alloys occurring in gases, in non-conductive liquids, or in most organic substances.
- Electrochemical corrosion – degradation occurring in electrolytic solutions or in the presence of moisture, accompanied by electric current flow through the metal.
- Atmospheric corrosion – caused by contact between metals and air pollutants.
- Biological corrosion – influenced by microorganisms that develop on metallic surfaces.

Chemical corrosion occurs when metals or alloys react at their surface with dry gases or non-electrolyte solutions. When exposed to electrolytic solutions, metals undergo electrochemical corrosion. Both the metal and the solution are traversed by an electric current produced by electrochemical reactions at the phase interface. Electrochemical corrosion requires the presence of an anode, a cathode, an electrolyte, and an electrical conductor. Impurities in the metal act as micro-cathodes, while the metal itself becomes the anode, dissolving into the surrounding liquid.

Atmospheric corrosion develops when a thin electrolyte layer forms due to humidity, enabling anodic and cathodic reactions on the metallic surface.

Microbiological agents capable of inducing corrosion include bacteria, fungi, and algae, which develop within a biofilm. Accumulated biofilms can form biodeposits several centimeters thick, significantly altering the chemistry at the metal–film interface.

Microorganisms contribute to corrosion primarily by accelerating electron transfer reactions.

Corrosion prevention may be achieved through protective coatings, use of corrosion inhibitors, polymeric layers, cathodic protection, or anodic protection.

Biocorrosion is driven by coordinated biochemical reactions constituting microbial metabolism. Oxidation–reduction processes generate a flux of chemical compounds through microbial cells.

The effect of bacteria on steels and aluminum alloys depends on the physicochemical parameters of the environment (aerobic, anaerobic, or mixed conditions). Under aerobic or anaerobic conditions, biocorrosion is localized at the biofilm–metal interface. In mixed environments, synergistic effects can lead to severe degradation.

The biocorrosion mechanism involves several pathways:

- production of corrosive metabolites;
- formation of microbial cell clusters;
- cathodic depolarization.

Microorganisms alter the chemical conditions of the storage environment, subsequently affecting the corrosion behavior of metallic materials.

In saline environments, corrosion results from electrochemical and adsorption phenomena arising from the interaction between salt crystals and peripheral metal grains. Ionic networks form through interactions between metallic atoms at the surface and NaCl ions.

In liquid media, the presence of dissolved impurities and high NaCl concentrations may occasionally inhibit corrosion due to deposition of limestone or carbonates, which create a protective barrier.

In microbiological environments, microorganisms act individually on metallic surfaces, influencing their mechanical response. Filamentous fungi produce organic acids that solubilize surface metal, decreasing plasticity. In some cases, microorganisms increase material plasticity by establishing electrochemical equilibria or by inducing the formation of ferric hydroxide precipitates. Although such mechanisms may locally improve mechanical resistance, AFM analysis reveals significant surface topography changes.

The deformability of the analyzed metallic materials (steel) depends strongly on chemical composition. Alloying elements from secondary subgroups promote

passivation, a chemical/electrochemical process that drastically reduces corrosion rate by forming a stable, adherent protective film.

Microbiological agents induce higher residual stress values in metallic materials (Al alloy), increasing the likelihood of crack initiation and propagation.

The 3^3 factorial mathematical models accurately describe the region in which the process attains optimal response values, though precision decreases near the boundaries of the chosen parameter domain.

Applying the 3^3 factorial design (pH, temperature, exposure time) indicates that a pH of 5-8, temperatures of 20-35 °C, and exposure times of 240-600 h (10-25 days) lead to favorable mechanical properties for metallic materials exposed to microorganisms.

ORIGINAL CONTRIBUTIONS

The theoretical and experimental research conducted in this thesis led to the following original contributions:

- Development of a comprehensive literature review on the action of various corrosive agents on metallic materials, with emphasis on microbiological corrosion. Comparison with saline corrosion revealed the influence of corrosive environment composition, temperature, and exposure duration on degradation mechanisms.
- Theoretical study of saline corrosion mechanisms, highlighting the role of Na^+ and Cl^- ions in destabilizing protective oxide films and generating stress concentrators.
- Detailed theoretical analysis of biocorrosion mechanisms for three steel types and one aluminum alloy, identifying the roles of microbial metabolic pathways, including anabolism and catabolism, and the formation of corrosive acidic by-products.
- Experimental evaluation of mechanical properties of metallic materials exposed to saline and microbiological environments (steel and Al alloy), using tensile–compression testing to quantify corrosion-induced degradation.
- AFM-based surface analysis demonstrating unfavorable surface alterations caused by metabolic by-products and corrosion deposits.

- Forming limit analysis (Marciniak method) showing the influence of chemical composition and microbial action on deformability.
- Residual stress analysis (hole-drilling method) demonstrating that microbiologically attacked Al alloy samples exhibit elevated tensile residual stresses, increasing the risk of premature failure.
- Application of the 3^3 factorial mathematical model to identify optimal pH, temperature, and exposure time ranges necessary to minimize biocorrosion effects and maintain favorable mechanical properties.

FUTURE RESEARCH DIRECTIONS

Further research may be pursued in the following directions:

- Analysis of additional environmental factors affecting metallic materials exposed to microbiological corrosion.
- Study of inhibitors targeting metabolic by-products involved in biocorrosion.
- Investigation of other corrosive environments and their effects on metallic material properties.
- Experimental testing of machining processes applied to biocorroded metallic components.
- Evaluation of other negative effects of corrosive agents (structural, chemical, microstructural modifications).
- Advancement of optimization techniques to define improved operational parameter domains for corrosion mitigation.
- Development of protective strategies to limit the action of various corrosive agents on metallic materials.

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