

Semi-Quantitative Categorization Method for the Corrosion Behavior of Metals Based on Immersion Test

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Abstract: Corrosion processes are complex in nature and their studies have become an interdisciplinary research field, combining fundamental sciences and engineering. As the quantification of corrosion processes is affected by many variables, standard guidelines to study such phenomena had been developed, such as ASME and ISO, and are broadly used in industry and academics. They describe methods to perform immersion test experiments and to quantify the corrosion rates of metals exposed to corrosive environments, but do not provide any guidelines for post-exposure analysis of the as-obtained corroded samples, which might provide useful information to understand the underlying physicochemical mechanisms of corrosion. This knowledge is useful for selecting optimal construction materials and developing corrosion prevention strategies. In this work, a semi-quantitative categorization method of the corrosion behavior of metals exposed to a corrosive medium based on their mass loss and aspect is presented. For each category, the mathematical aspects of gravimetric measurements of mass change rate and the analytical techniques that can be used for the characterization of materials are discussed. The following method does not intend to replace industrial standards, but to expand them in order to maximize the amount of information that can be extracted from immersion tests.

Keywords: corrosion type; immersion test; corrosion mechanisms; corrosion rate quantification; corrosion behavior; semi-quantitative categorization method



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1. Introduction

The phenomenon of corrosion represents a natural process wherein a refined metal undergoes transformation into a more chemically stable form, such as its oxide, hydroxide, or sulfide. This gradual deterioration of materials, primarily metals, results from chemical and/or electrochemical reactions with their environment, leading to the degradation of essential material properties, including strength. The repercussions of corrosion are significant, as it can accelerate structural and equipment failures in both domestic and industrial applications, such as those within the oil, gas, and chemical industries, thereby potentially causing catastrophic consequences [1]. To avert such situations, substantial financial resources, amounting to billions of dollars annually worldwide, are allocated to combatting corrosion. For instance, a comprehensive study spanning from 1999 to 2001 estimated the annual direct cost of corrosion in the United States to be USD 276 billion, constituting approximately 3.1% of the nation's gross domestic product (GDP) [2].

The evaluation of material durability in reactive environments frequently involves corrosion tests, with electrochemical and gravimetric methods being the primary approaches. While this work does not delve into electrochemical methods, gravimetric techniques, particularly the mass loss test or immersion test, are widely employed to study corrosion. The NACE TM0169/G31-12a “Standard Guide for Laboratory Immersion Corrosion Testing of Metals” [3], established by corrosion specialists in industrial processes, outlines the best practices for conducting corrosion tests through this method. The standard comprehensively addresses factors influencing laboratory immersion corrosion tests, result

interpretation, and corrosion rate calculations. Despite acknowledging the complexity of corrosion phenomena and variations in how metals respond to influencing factors, the standard provides valuable guidance to minimize errors and interferences in determining corrosion rates.

After the immersion test, the standard advises conducting a visual inspection of the sample and subsequently implementing rigorous cleaning methods (mechanical, chemical, or electrochemical) to eliminate all corrosion products from the specimens for the purpose of quantifying corrosion rates. Nevertheless, this conventional approach poses the risk of losing critical information, including the chemical nature and morphology of the chemical species formed on the metal. In this paper, an alternative methodology is proposed wherein, prior to removing corrosion products, the sample undergoes a sequence of cleaning, drying, and weighing. The magnitude of the mass change without the removal of corrosion products, along with the sample's aspect, can then classify it into one of five specified categories outlined in this study. Importantly, this approach advocates for maximizing the information obtained through immersion test experiments by postponing the removal of corrosion products until after the initial examination and assessment.

1.1. Corrosion Behavior Classification

Efforts have been made to establish classification and prediction models for the corrosion behavior exhibited by metals under varying corrosive environments. The predominant focus of these endeavors lies in electrochemical methods, whereas gravimetric measurements are tailored to specific alloy types or corrosive settings.

Various corrosion type classification methods based on electrochemical noise techniques, including statistical analysis and Fourier transform approaches, have been employed to investigate corrosion rate and type [4–6]. Parameters such as current standard deviation, voltage standard deviation, noise resistance, and power spectral density (PSD) diagrams have been examined in various works [7,8]. In the realm of advancing technologies, researchers have introduced novel electrochemical noise data analysis methods, including cluster analysis, linear discrimination, neural networks, and gradient-boosting decision trees [5]. One study presented an electrochemical noise analysis method based on Adaboos and identified three categories for the corrosion behavior of metals: passivation (0), uniform corrosion (1), and pitting corrosion (2) [5]. In recent years, there has been a growing interest in employing machine learning techniques to classify corrosion based on alternative electrochemical measurements, including the Tafel extrapolation method [9–15].

Approaches for classifying corrosion behaviors based on gravimetric methods predominantly rely on the quantification of corrosion rates (CR) as a foundational measure. These methods are generally tailored to specific alloy types and corrosive environments, lacking a universally applicable methodology across diverse sectors. An example of recent efforts involves the classification of metals exposed to seawater, employing an immersion test that categorizes corrosion into six levels (C1 (very low) to CX (extreme)) based on penetration rates [16]. Moreover, contemporary research has explored the integration of machine learning techniques to predict corrosion behavior using CR as a key parameter. In a particular study, algorithms were trained based on defined criteria: resistant ($\text{MLR} \leq 0.1 \text{ g.h}^{-1}.\text{m}^{-2}$ or $\text{CR} \leq 0.11 \text{ mm/y}$), good ($0.1 < \text{MLR} \leq 1.0 \text{ g.h}^{-1}.\text{m}^{-2}$ or $0.11 < \text{CR} \leq 1.10 \text{ mm/y}$), poor ($1.0 < \text{MLR} \leq 10.0 \text{ g.h}^{-1}.\text{m}^{-2}$ or $1.10 < \text{CR} \leq 11.0 \text{ mm/y}$), and severe ($\text{MLR} > 10.0 \text{ g.h}^{-1}.\text{m}^{-2}$ or $\text{CR} > 11.0 \text{ mm/y}$) [15].

This study aims to introduce a categorization method, utilizing gravimetric techniques, that is applicable to the corrosion behavior of metals exposed to various corrosive environments, with broad applicability across different systems.

1.2. Corrosion Rates

The primary methods employed in corrosion studies include gravimetric methods and electrochemical methods, the latter of which will not be discussed in this context. Gravimetric methods, the predominant technique for practical applications, involve mea-

suring mass loss by immersing a metal sample in a corrosive medium (immersion test). This well-established method determines the weight difference of the specimen before and after immersion for a specified duration [17]. However, this method presents noteworthy limitations [17]:

1. It provides only the average corrosion rate over the immersion test duration, lacking the capability to discern differences in oxidation kinetics;
2. Inaccuracies in corrosion rate determination may arise due to inadequate or excessive removal of corrosion products post-immersion;
3. It does not offer insights into the underlying corrosion mechanisms.

Corrosion, fundamentally an electrochemical process, makes electrochemical methods a potent tool for studying metal corrosion in corrosive environments [17]. These methods, such as the Tafel extrapolation method, can estimate the corrosion rate (CR) and assess the efficacy of corrosion protection methods. However, detailed corrosion mechanisms require a variety of complementary techniques [17]. In essence, gravimetric methods for quantifying corrosion rates are straightforward and do not involve measuring currents or voltages. On the other hand, electrochemical techniques, like Tafel extrapolation, demand high anodic/cathodic polarizing voltages (e.g., ± 250 mV) and may alter electrode surface properties [18], rendering the results non-representative of real conditions in industrial plant construction materials. Consequently, this work focuses on studying corrosion phenomena using gravimetric methods, reserving electrochemical methods for studying fundamental corrosion mechanisms and trend evaluation.

1.3. Standard Methods for Corrosion Rate Quantification via Immersion Test

This section provides a concise overview of the primary method outlined in the literature for quantifying corrosion rates through immersion experiments. Widely employed industry and academic guidelines, such as ISO, ASME, and NACE TM0169/G31-12a “Standard Guide for Laboratory Immersion Corrosion Testing of Metals” [3], offer comprehensive instructions for conducting immersion test experiments and determining corrosion rates for metals exposed to corrosive environments. Despite their broad usage, these standards have notable limitations, particularly the absence of guidelines for the post-exposure analysis of obtained corroded samples. Post-exposure analysis is crucial for gaining insights into the physicochemical mechanisms of corrosion, although it is not the primary focus of these standards. This information is, however, valuable for selecting appropriate construction materials and developing effective corrosion prevention strategies [19,20].

Upon completion of the immersion test, the recommended procedure includes a visual inspection of the metal sample, followed by thorough cleaning (mechanical, chemical, or electrochemical) [3]. This cleaning process is aimed at removing all corrosion products from the specimens to quantify the corrosion rate by measuring the mass difference of the corrosion-products-free corroded sample. However, this approach has a significant drawback as it results in the loss of vital information, such as the chemical nature and morphology of the chemical species forming on the metal.

Standard ISO 11845:2020, titled “Corrosion of metals and alloys-General principles for corrosion testing,” also provides guidelines for evaluating corrosion rates through immersion [10]. The gravimetric method involves measuring the mass loss of a material with known dimensions when immersed in a corrosive media for a specified duration. The weight of the specimen is recorded before and after exposure, with procedures outlined in ASTM G 31, “Standard Guide For Laboratory Immersion Corrosion Testing of Materials” [3], and Standard ISO 11845:2020 [21].

Both standards require the removal of corrosion products from the surface to quantify corrosion rates, referencing cleaning procedures (ASTM G1 [22] and ISO 8407 [23]) designed to eliminate corrosion products while minimizing the removal of sound metal. Notably, ISO 11845:2020 recommends weighing specimens before corrosion product removal, calculating the mass of corrosion products obtained by difference, but does not establish specific

protocols for washing to ensure the removal of the corrosion media without disturbing the corrosion products.

2. Method

2.1. Proposed Method

In this methodology, a modified version of the standard method is employed to investigate the corrosion products and morphological characteristics of the metal surface post-exposure, with the aim of enhancing the information derived from the immersion test and classifying the systems based on behavioral types as described in Section 3. The modification is outlined as follows:

Prior to the removal of corrosion products from the specimens, in accordance with the ASTM G1 [22] and ISO 8407 [23] standards, a pre-cleaning step is performed using solvents in which the corrosion products are expected to be insoluble and exhibit minimal reactivity with the metal, such as acetone, alcohol, and water. Abrasive materials and ultrasound should be excluded from the cleaning process to mitigate the potential loss of corrosion products from the metal surface. The cleaning procedure involves immersing the coupons in various solvents, including acetone, alcohol, and water, followed by gentle rinsing. These steps are repeated as necessary to eliminate the corrosive medium. Upon completion of the cleaning process, the samples are allowed to air-dry in a desiccator. Towels should be avoided during the drying phase to prevent potential errors arising from specimen contamination with grease or lint and to preserve corrosion products. Test specimens should be handled with gloves and plastic tweezers to minimize surface contamination after cleaning.

Following the cleaning procedure described above, the masses of the dried, corroded test specimens are measured with an accuracy corresponding to that of the original mass measurements, aligning with the recommendations in ISO 11845:2020 [21]. The mass of loose corrosion products that detached and fell from the metal surface into the weighing boat or paper is considered when quantifying the final mass of the metal coupon. The mass loss or gain during the test period, along with the aspect of the sample, are then utilized to classify the system according to the method presented in Section 3.

2.2. Gravimetric Methods-Mass Balance

The mass balances for determining the corrosion rate through gravimetric methods are expressed as follows:

$$M_{\text{ini}} = M_{\text{metal-final}} + M_{\text{solution}} + M_{\text{losses}} + O_{\text{ini}} \quad (1)$$

$$M_{\text{final}} = M_{\text{metal-final}} + M_{\text{CP}} + O_{\text{CP}} \quad (2)$$

$$\Delta m_{\text{MCR}} = M_{\text{ini}} - M_{\text{final}} \quad (3)$$

$$\Delta m_{\text{CR}} = M_{\text{ini}} - M_{\text{metal-final}} \quad (4)$$

where:

M_{ini} is the initial mass of the tested material.

M_{final} is the final mass of the cleaned and dried tested material after the immersion test, including the remaining metal ($M_{\text{metal-final}}$) and any metal present in the corrosion products deposited on the surface (M_{CP}).

M_{solution} is the metal content in the solution, calculated from the total solvent amount multiplied by the metal concentration determined by analytical techniques such as inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma optical emission spectrometry (ICP-OES), or ion chromatography (IC).

M_{losses} represent the metal present in corrosion products that did not grow on the metal surface, or in corrosion products that fell during the immersion test or washing steps, and metal that might be adsorbed into the surfaces of containers and equipment.

O_{ini} represents the mass of other elements in the original sample, excluding the metal of interest, such as oxygen from oxide layers. It can be reduced to zero through pre-testing cleaning procedures. O_{CP} denotes the mass of other elements incorporated into corrosion products in the sample, either initially present in the sample or derived from the corrosive environment, contributing to the final sample mass.

The mass change for calculating the mass change rate (MCR), Δm_{MCR} , is given by Equation (3). This value can be positive if the material is dissolving at a higher rate than corrosion products (if any) are depositing on the metal surface, or negative if corrosion products are growing significantly on the material surface. The MCR can be calculated using Equation (5).

For the calculation of the corrosion rate (CR), according to ASTM G 31 [3] and ISO 11845:2020 [21] standards, Equation (4) is used. Further treatment of the sample may be necessary to selectively remove corrosion products (M_{CP}), leaving the sound metal ($M_{metal-final}$). This equation should always yield positive values for CR, as the mass change (Δm_{CR}) in these standards corresponds to the cleaned sample after removing corrosion products.

After weighing the sample containing corrosion products (M_{final}), it can be categorized using the method developed in Section 3 and analyzed with non-destructive tests. After that, corrosion products can be mechanically removed according to the methods described in the ASTM G1 [22] and ISO 8407 [23] standards, and corrosion rates can be quantified following the ASTM G 31 [3] and ISO 11845:2020 [21] standards (Equation (6)). Figure 1 illustrates the proposed methodology.

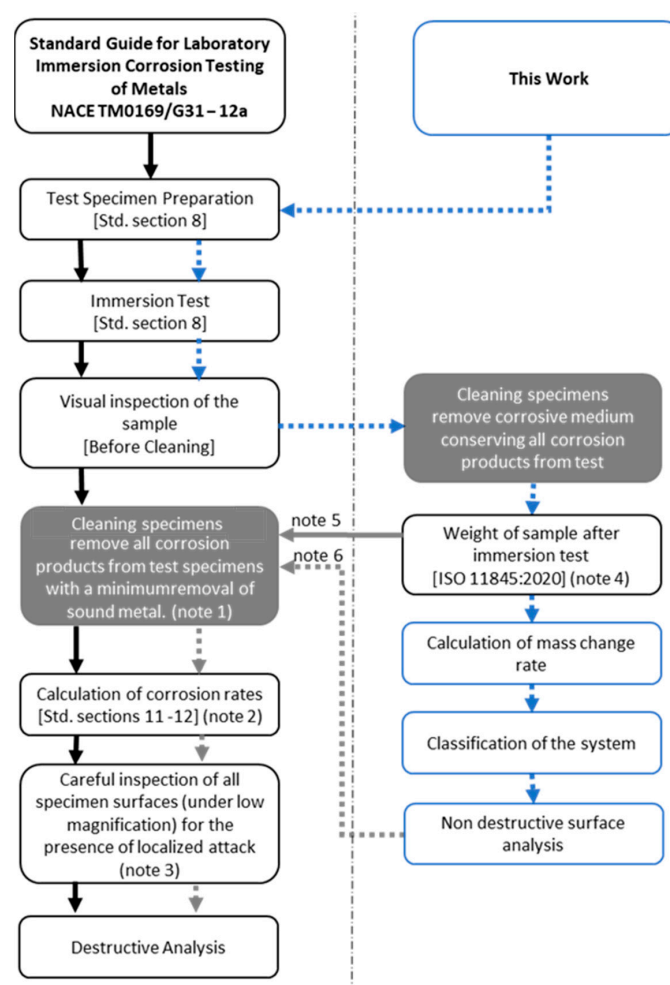


Figure 1. Main steps to quantify the corrosion rates. Note 1. ASTM Practice G1 (Section 10) and ISO 8407. Note 2. If the variance between samples exceeds 10%, the causes of the reason for the disparity

should be investigated and reported. If the reason for the disparity cannot be found, retesting should be considered. Note 3. If pits are observed, they should be evaluated following the guidance of ASTM Guide G46. Note 4. Standard ISO 11845:2020 recommends that specimens should also be weighed before corrosion product removal and the mass of the corrosion products should be obtained by difference. Note 5. The path indicated in ISO 11845:2020. Note 6. Proposed path to continue the determination of the corrosion rates as per current standards. Reprinted from Ref. [24].

2.3. Corrosion Rate Quantification

Corrosion rates (CRs) are typically denoted as penetration rates (units of length/unit of time). The proposed mass change rate (MCR) calculation, as presented here, is not expressed in these traditional units due to the potential impact of corrosion products on the overall and/or local density of the material (Equation (5)). In certain cases, CR has been expressed solely as a penetration rate for guidance (Equation (6)). Discrepancies between MCR and CR can be utilized to estimate the kinetics of corrosion product formation on the material surface.

$$\text{MCR} = \frac{\Delta m_{\text{MCR}}}{A \cdot t} \quad (5)$$

$$\text{CR} = \frac{k \cdot \Delta m_{\text{MCR}}}{A \cdot t \cdot \rho} \quad (6)$$

where Δm_{MCR} is the mass loss or gain [mg] by the metal in time t [d]. ρ is the density of the material in $[\text{g}/\text{cm}^3]$, k is a constant (8.76×10^4 , ensuring CR is in mm/y), and A is the surface area of the exposed material $[\text{cm}^2]$.

It is important to note that Equations (5) and (6) consider the geometrical initial area without accounting for roughness. Additionally, these equations assume that the surface area remains constant throughout the corrosion process. Previous research [25] has demonstrated that this assumption underestimates the true CR, and significant errors can arise if changes in area are not considered, particularly for 3D and rod-like objects. Errors in the CR calculation have the potential to lead to the premature failure of metallic structures designed with inaccurate data. Accurate CR determination can be achieved by solving cubic equations that account for changes in area [25]. The errors associated with assuming constant areas for sheet-like objects are minimal, and Equation (6) can be applied. However, conducting corrosion testing on thin films can be experimentally challenging, as metal foils lack rigidity and can easily break. The cubic equations for disks (Equation (7)) and rectangular geometries (Equation (8)) under active dissolution (generalized corrosion) are as follows:

$$-2\pi \cdot \text{CR}^3 \cdot t^3 + \pi(2D_0 + h_0) \cdot \text{CR}^2 \cdot t^2 - \frac{\pi}{2}(D_0^2 + 2D_0 \cdot h_0) \cdot \text{CR} \cdot t - \frac{\Delta m}{\rho} = 0 \quad (7)$$

$$-8 \cdot \text{CR}^3 \cdot t^3 + 4(2x_0 + z_0) \cdot \text{CR}^2 \cdot t^2 - 2(x_0^2 + 2x_0 \cdot z_0) \cdot \text{CR} \cdot t - \frac{\Delta m}{\rho} = 0 \quad (8)$$

where D_0 and h_0 are the initial diameter and height of a cylinder (or disk), and X_0 , Y_0 , and Z_0 are the initial dimensions for rectangular prisms and sheets. Details on Equations (5) and (6) and a spreadsheet are included in the Supplementary Material in [25].

2.4. Surface Analysis

The impact of surface features, including texture, surface energy, and mechanical processing-induced defects, on corrosion behavior is widely acknowledged in the academic literature [3]. A comprehensive understanding of corrosion processes necessitates the characterization of material surfaces. Various methods for surface analysis, including both in situ and ex situ post-exposure, along with their operational principles, measuring techniques, applications, and the advantages and limitations of commonly-used techniques for corrosion characterization and electrochemical studies, have been reviewed and are

summarized in Table A1 Appendix A [26–29]. These methods encompass a range of techniques, such as atomic force microscopy (AFM), X-ray diffraction spectroscopy (XRD), high-temperature XRD, X-ray photoelectron spectroscopy (XPS) [26], Raman spectroscopy, and Fourier transform infrared spectroscopy (FTIR).

Additionally, emerging techniques for surface characterization, as outlined in the literature [19,20], include Kelvin probe force microscopy (KPFM), scanning tunneling microscopy (STM), scanning electrochemical microscopy (SECM), secondary ion mass spectrometry (SIMS), X-ray absorption spectroscopy, Rutherford backscattering spectroscopy, Auger electron spectroscopy, particle-induced X-ray emission [26], electron probe microanalysis (EPMA), near-edge X-ray absorption fine structure spectroscopy, X-ray photoemission electron microscopy, low-energy electron diffraction, small-angle neutron scattering, neutron reflectometry, and conversion electron Mössbauer spectroscopy (CEMS).

Several of these techniques are non-destructive and can be employed post-corrosion to identify corrosion products on the surface. For example, XRD can identify crystalline phases, whereas XPS can reveal oxidation states. Other non-destructive methods like X-ray fluorescence (XRF) can determine the bulk chemical composition, which is particularly useful for studying selective demetallation in alloys or detecting the presence of the corrosive medium in corrosion products or material inclusions.

As more materials are categorized, it is anticipated that the most suitable analytical methods from the mentioned techniques, including emerging surface analysis techniques, will be identified for each system type.

The NACE standard mentions 3 post-exposure tests that can be carried out after the immersion test: (1) mechanical property comparison (tensile strength) ASTM Test Methods E8. (2) Cross-section microscopical examination can be used to assess dealloying or intergranular attacks, and (3) simple bending tests to determine whether any embrittlement attack has occurred.

3. Semiquantitative-Categorization System for the Corrosion Behavior of Metals Based on Immersion Tests

In this methodology, the corrosion behavior of metals exposed to corrosive media is categorized into different types. Following the cleaning of the sample without removal of corrosion products, the calculation of MCR, and the visual inspection of the metal sample, the flow diagram (Figure 2) serves as a classification tool.

Immune metals (type-0) exhibit no reactivity with the corrosive medium, making them ideal construction materials with excellent corrosion resistance.

Passive metals, in a passive state, are characterized by a continuous “passivating film” of solid corrosion products that separates the metallic phase from the adjacent corrosive medium. Corrosion in the passive state involves the growth of the passivating film and/or the transfer of metal ions through the film into the corrosive medium [30]. A metal is considered passive if it significantly resists corrosion in a given environment despite a marked thermodynamic tendency to react [31]. Passive metals are further classified into type-1 (forming thin films, resulting in a negligible mass change in the specimen as subsequently defined), type-2S, or type-3S (forming thick films). Passive metals can be used as construction materials under specific conditions.

Active metals, in an active state, undergo corrosion through the direct transfer of metal ions from the metallic phase to the adjacent corrosive medium [30]. In the context of this categorization method, an active metal is either transformed into corrosion products without forming protective passive layers (type-2 and type-3) or actively dissolves into the corrosive medium (type-4). Materials in this category are unsuitable as construction materials.

Figure 3 illustrates hypothetical curves depicting the mass change as a function of time for each corrosion behavior classification type.

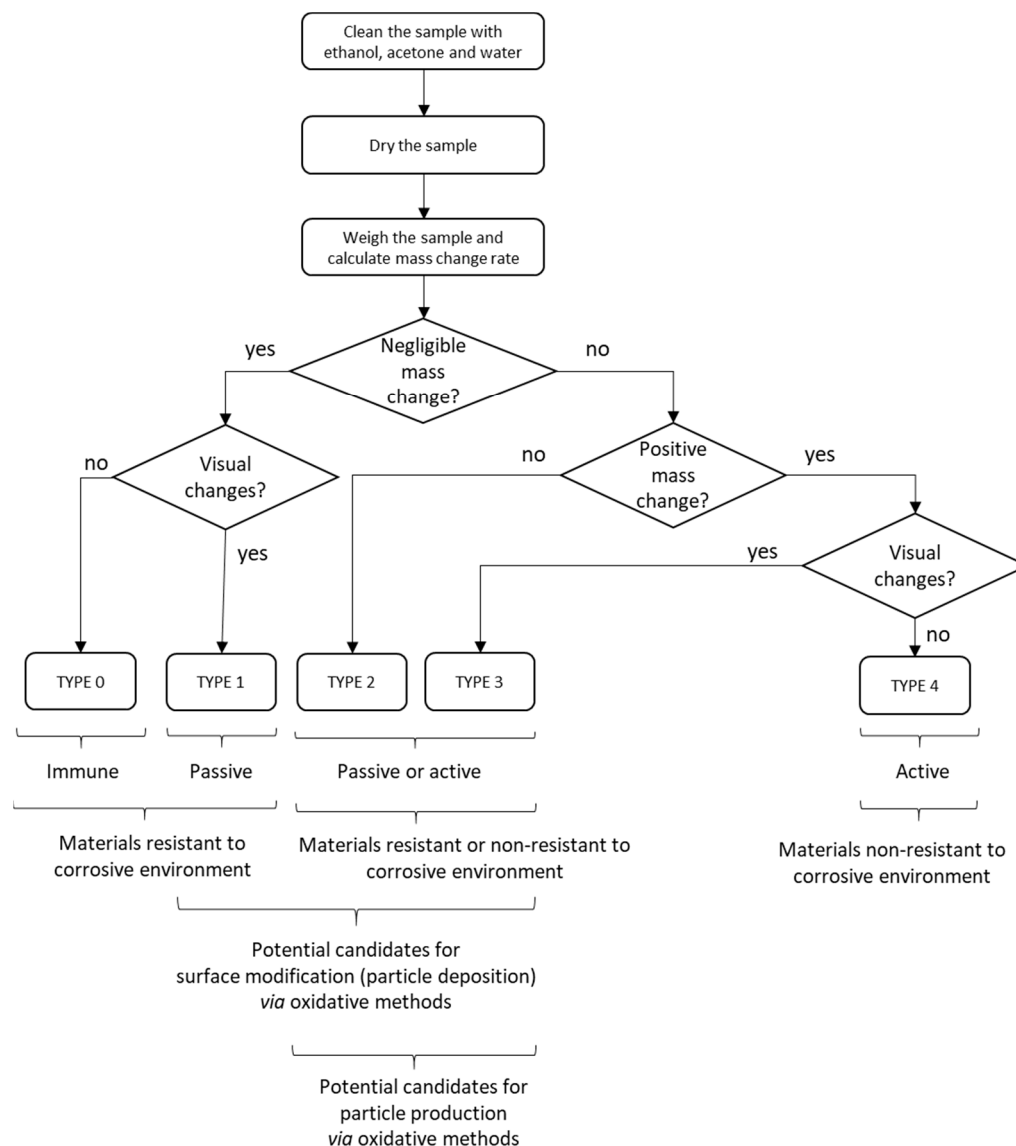


Figure 2. Flow diagram used to classify the corrosion behavior following the immersion test according to the semi-quantitative categorization method. Reprinted from Ref. [24].

Negligible mass changes: When the magnitude of the corrosion rate is extremely small and approaches the precision limit of the quantification method (i.e., precision of the balance and area determination), it becomes challenging to determine accurately. The collected MCR data exhibit significant scatter, stemming from errors in the weighing step. The resulting mass change rate is so minimal that the CR is less than 0.05 mm/y. Retesting a sample at longer exposure times could be considered to confirm this behavior, especially if the exposure times were initially short.

Passivation: In this context, passivation refers to a material experiencing reduced corrosion with increasing exposure times. Passivation involves the formation of a stable outer layer of shielding material through spontaneous chemical reactions with both the material and the corrosive environment. For instance, materials like lead, when exposed to sulfuric acid, initially corrode at a high rate while forming a protective film. Subsequently, the corrosion rate decreases considerably, rendering further corrosion negligible.

Visual changes: Visual changes denote variations in the appearance of the sample before and after the immersion test. This includes alterations in color or the formation of corrosion products deposited on the metal surface, which persist after the cleaning process. If corrosion products are observed in the reaction medium but not on the metal

surface, or if they are easily removed during cleaning, it can be inferred that there are no significant changes. In some cases, when using raw surfaces without prior cleaning before the immersion test, metals may appear shiny after exposure to the corrosive medium. This shine could result from the dissolution of an oxide layer, similar to a chemical cleaning. For the classification system's purposes, in such cases, this can be considered as no visual changes. Morphological changes on the surface, detectable at high magnifications, do not meet the criteria for visual changes.

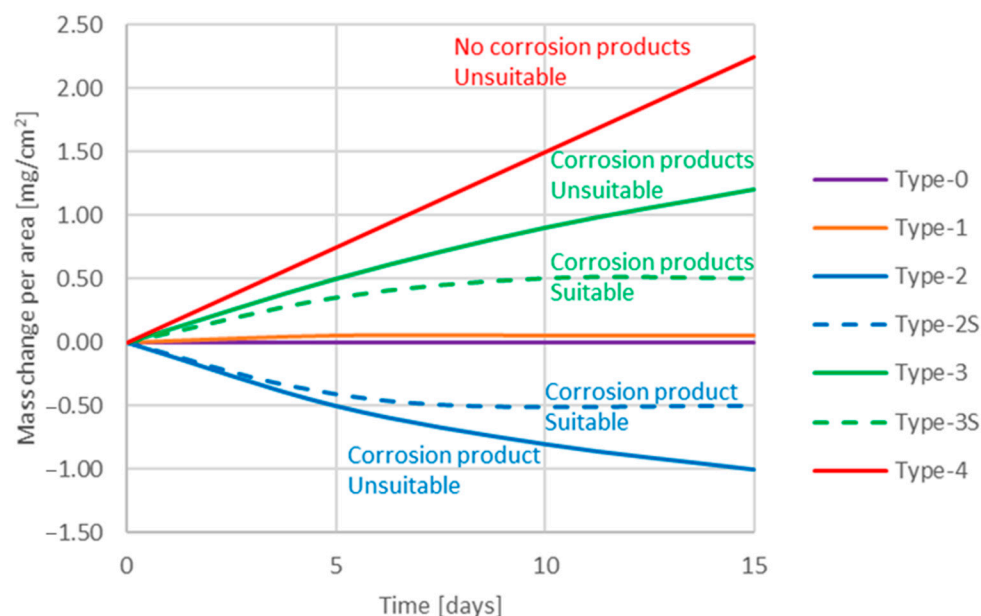


Figure 3. Hypothetical curves illustrating the mass change as a function of time for each corrosion behavior classification type. Reprinted from Ref. [24].

3.1. Categories

3.1.1. Type 0

This category represents the ideal scenario, where a material remains unaffected by its environment. Materials classified as type 0 exhibit a negligible mass change rate during the immersion test method. Visual inspection cannot distinguish an exposed piece from an untreated one (Figure 4). Detailed surface analyses, such as SEM, XRF, or EDX, are also unable to differentiate between untreated samples and those in this category.

For metals classified as type 0, mechanical cleaning is unnecessary, and the MCR (divided by the density) and the CR should be identical. The metal content in the solution, as well as metal losses (M_{loss}), are negligible and likely to fall below the detection limits of common instrumental analyses.

It is probable that these systems are not influenced by the area-to-solvent ratio. Therefore, smaller quantities than those recommended in ASTM G 31 [3] and ISO 11845:2020 [21] standards may be sufficient, especially if the solvents are challenging to synthesize or expensive.

3.1.2. Type 1

Type 1 materials, similar to type 0, exhibit an extremely small MCR that may be challenging to accurately quantify using the immersion test method. However, noticeable changes in the metal's appearance, such as color changes, are evident (Figure 4). Materials in this category rapidly form stable passive layers, providing protection against further corrosion. As these layers are very thin, overall mass changes are minimal. Typically, samples under this category are indistinguishable from untreated samples in more detailed surface analyses such as SEM, XRF, or EDX. Additional techniques for thin-film analysis, like XPS, are necessary to study the nature of the corrosion products.

For type 1 materials, it is anticipated that the CR is slightly higher than the MCR (divided by the density) due to the removal of the protective layer during the cleaning process. The metal content in the solution, as well as metal losses (M_{loss}), are negligible and likely to fall below the detection limits of common instrumental analyses.

As with type 0 metals, it is probable that these systems are not significantly affected by the area-to-solvent ratio. Therefore, smaller quantities than those recommended in ASTM G 31 [3] and ISO 11845:2020 [21] standards may be sufficient, especially if the solvents are challenging to synthesize or expensive.

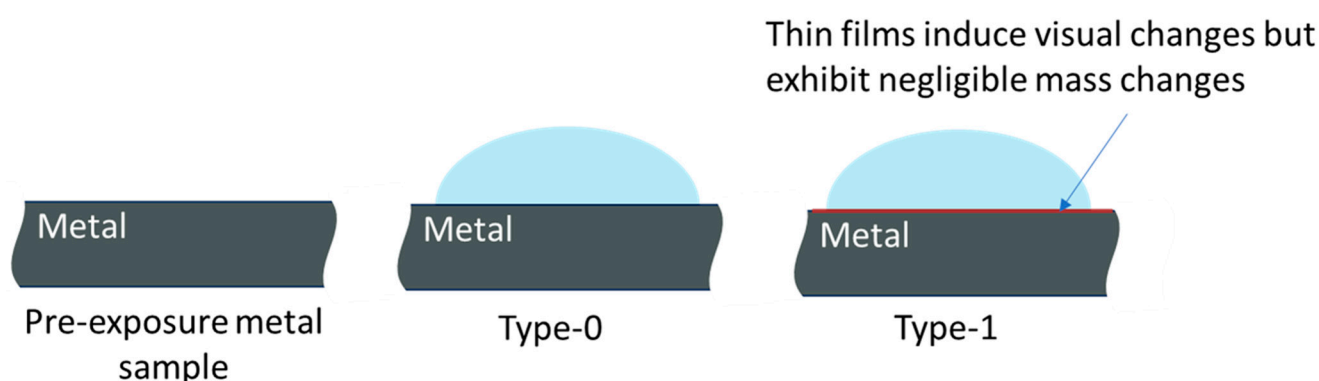


Figure 4. Scheme depicting the corrosion behavior of type 0 and type 1 metals, characterized by negligible mass change rates and $CR < 0.05$ mm/y.

3.1.3. Type 2

Materials in this category undergo active anodic reactions with the corrosive medium, leading to the formation of corrosion product layers that result in a net mass gain. In these systems, the corrosion products exhibit very low solubilities in the corrosion medium, and their crystals firmly grow on the metal surface. System passivation is possible in these cases (type 2S). If the formed layers do not provide sufficient protection, the attack will persist until the material is ultimately compromised. Changes in these systems are typically detectable by SEM, XRD, EDX, or XRF, depending on the nature of the products (Figure 5).

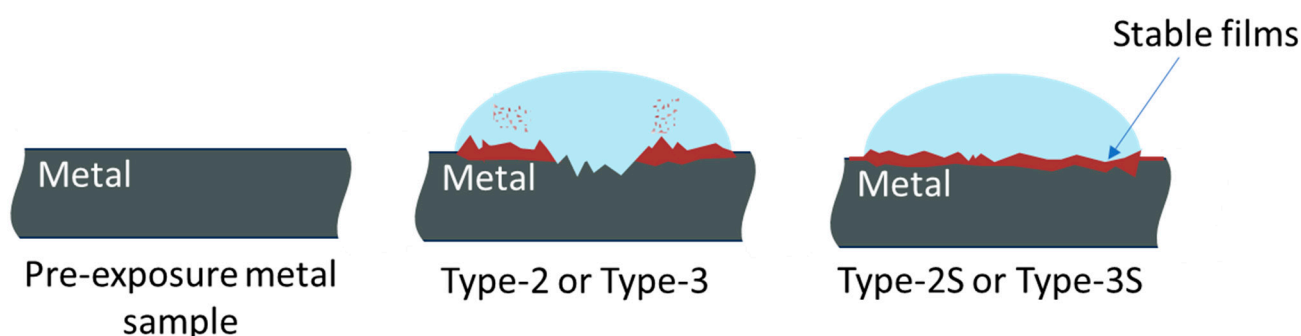


Figure 5. Scheme showing the corrosion behavior of type 2 (or type 3) and type 2S (or type 3S) metals. Type 2 and type 3 materials exhibit $CR > 0.05$ mm/y with the formation of visible corrosion products on the surface without passivation. Type 2S and type 3S exhibit stable corrosion product films. Type 2 materials exhibit a net mass gain, whereas type 3 materials exhibit a net mass loss.

The use of type 2 materials as construction materials is not recommended unless the material forms a stable passive layer (type-2S). Mechanical cleaning is necessary for CR quantification, with $CR > MCR$ (divided by the density) due to the presence of deposition products. Similar to type 3 materials, the metal concentration in the corrosion medium depends on the solubility of the corrosion products in the solvent and the rate of formation of corrosion products. In these systems, solvents may or may not reach saturation,

depending on the amount of material used. Significant losses may occur, as the crystals can grow in the bulk solution or other places and may detach during the immersion test or cleaning step.

As the corrosion phenomenon occurs at the interphase between the material and the medium, the presence of corrosion products being deposited on the surface is expected to modify the effective area. Therefore, measurements of MCR and CR for type 2/2S and type 3/3S materials are likely to be less accurate.

3.1.4. Type 3

Similarly to type 2 materials, metals in this category undergo active anodic dissolution in the corrosive medium, leading to the formation of corrosion products on the surface; however, samples experience a net mass loss. In these systems, passivation is possible (type-3S). If the formed layers do not provide sufficient protection, the attack will persist until the material is ultimately compromised. In these systems, the corrosion products exhibit low-partial solubilities in the corrosive medium, and their crystals might nucleate and grow in the bulk solution as well as on the surface of the metal. Changes in these systems are typically detectable by SEM, XRD, EDX, or XRF, depending on the nature of the products.

The use of type 3 materials as construction materials is not recommended unless the material forms a stable passive layer (type 3S). Mechanical cleaning is necessary for CR quantification, with $CR > MCR$ (divided by the density) due to the presence of deposition products. The metal concentration in the corrosion medium depends on the solubility of the corrosion products in the solvent and the rate of formation of corrosion products. In these systems, solvents may or may not reach saturation, depending on the amount of material used. Significant losses may occur, as crystals might grow in the bulk solution or other places and can detach during the immersion test or cleaning step. Corrosion products are loosely adherent to the surface and do not form protective films.

As the corrosion phenomenon occurs at the interphase between the material and the medium, the presence of corrosion products being deposited on the surface is expected to modify the effective area. Therefore, measurements of MCR and CR for these kinds of materials are likely to be less accurate.

3.1.5. Type 4

Materials in this category undergo active anodic dissolution in the corrosive medium without the formation of corrosion products or passive layers (generalized corrosion) (Figure 6). In these systems, corrosion products exhibit high solubilities in the corrosive medium. Typically, no changes are detected by XRD, EDX, or XRF. However, in some instances, changes in the morphology of the surface can be detected by SEM.

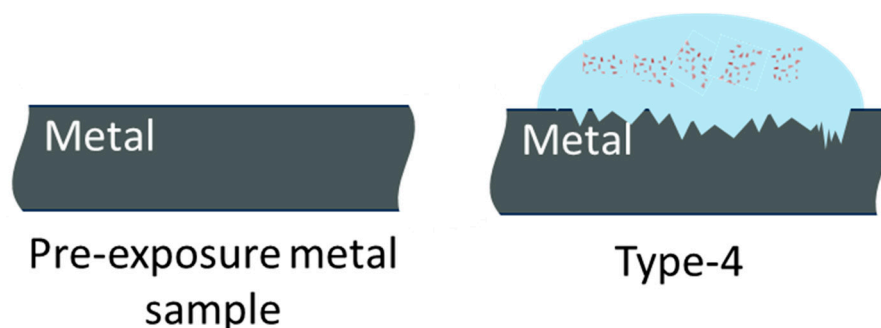


Figure 6. Scheme showing the corrosion behavior of type 4 metals. In these systems, layers of corrosion products are not formed over the surface of the metal.

The use of type 4 metals as construction materials is strongly discouraged. Mechanical cleaning for CR quantification is not required, and the MCR and the CR should be identical,

as no corrosion products are forming on the surface. The metal concentration in the corrosion medium is significant. The material mass change should be identical to the metal content in the solution if corrosion products are not growing in the bulk solution nor being adsorbed by the wetted parts of the corrosion-testing apparatus.

3.1.6. Suitable Materials (S-Suffix)

Materials in this category undergo active anodic dissolution with the formation of stable protective layers that offer sufficient protection against corrosion (system passivation). Therefore, these materials might be suitable for use as construction materials. This behavior is characterized by either an initial mass gain (type-2S) or an initial mass loss (type-3S), followed by a steady-state condition.

3.1.7. Summary

Tables 1 and 2 provide a summary of the key characteristics of the semi-qualitative classification system for the corrosion behavior of metals exposed to corrosive media.

Table 1. Summary of the criteria for the semi-quantitative categorization system.

Type	MCR	MCR Magnitude	Visual Changes	Passivation	Corrosion Product *	CR-MCR ** Relationships
0	Negligible	$\pm$	No	n/a	No	CR = MCR
1	Negligible	$\pm$	Yes	Yes	Yes	CR $\geq$ MCR
2	Significant	-	Yes	Possible	Yes	CR > MCR
3	Significant	+	Yes	Possible	Yes	CR > MCR
4	Significant	+	No	No	No	CR = MCR

* Insoluble corrosion products formed on the surface. ** MCR divided by the density.

Table 2. Summary of the mass balance parameters.

Type	M_{final}	$M_{\text{metal-final}}$	M_{CP}	M_{solution}	M_{losses}
0	$\approx M_{\text{ini}}$	$\approx M_{\text{ini}}$	0	0	0
1	$\approx M_{\text{ini}}$	$\approx M_{\text{ini}}$	≈ 0	≈ 0	≈ 0
2	$> M_{\text{ini}}$	$< M_{\text{ini}}$	> 0	$> \text{or}$	$> \text{or} \approx 0$
3	$< M_{\text{ini}}$	$< M_{\text{ini}}$	> 0	$> \text{or} \approx 0$	$> \text{or} \approx 0$
4	$< M_{\text{ini}}$	$< M_{\text{ini}}$	0	$> \text{or} \approx 0$	$> \text{or} \approx 0$

4. Classification Method Disambiguation

As emphasized in the ASTM G 31 [3] standard and other references [32], corrosion processes are intricate and influenced by numerous variables. Given that the corrosion behavior of materials exposed to a corrosive environment can be impacted by experimental conditions, it is crucial to meticulously document relevant factors and conditions for accurate result interpretation. These factors encompass heat transfer (temperature differentials between the metal and corrosive environment), fluid motion, corrosion product formation, specimen nature (e.g., chemical composition, presence of welds, metal sample preparation, cast and wrought alloys), and solution properties (e.g., composition, dissolved oxygen, impurity concentration, temperature, and pH value).

Impact of geometry: The specimen's shape may affect the inferred behavior type. For instance, employing thin samples minimizes errors in CR or MCR calculations related to the assumption of a constant area. However, thin samples are challenging to measure and handle, and they may succumb to corrosion before stable passive layers form. If passivation layer formation (materials type-2S and type-3S) is suspected, samples with substantial thickness should be used (Figure 7 path (a)).

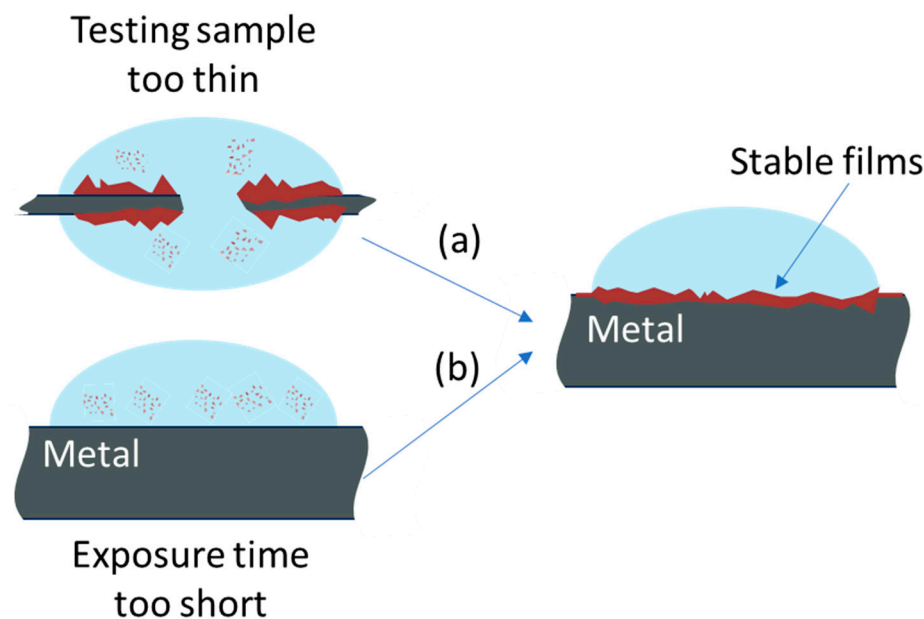


Figure 7. Illustration demonstrating potential scenarios leading to misclassification. Path (a) depicts a thin sample collapsing before stable films form to passivate the surface, potentially misinterpreted as type 4 material. Retesting with optimal thickness would correct the categorization to type 2S/3S. Path (b) illustrates slow nucleation kinetics, preventing critical concentration attainment for stable film growth. Initial results may suggest type 3 or 4 categorization, but retesting at extended times would correctly classify it as type 2S/3S.

Impact of exposure time: The duration of the corrosion test can influence the system's category, necessitating the explicit mention of the test's duration. Materials might be classified differently at short and long exposure times. For example, a short test of a material exhibiting passivation could yield an initially high corrosion rate, potentially misleading interpretations. Additionally, at short exposure times, there may not be sufficient corrosion product formation (due to slow kinetics), leading to a different classification (Figure 7 path (b)). The ASTM G 31 2 standard recommends longer tests for more realistic results, cautioning against corrosion reaching points of drastic specimen size reduction, exposed area reduction, or perforation.

The author contends that, with the guidelines presented in this work, systems can generally be categorized into one type. However, some systems may exhibit borderline behaviors. In such cases, it may be indicated that the tests are inconclusive for categorizing the system or that suspected categories are mentioned, along with reasons why a single category could not be assigned. Retesting the specimen with different conditions, such as altered geometry, solvent-to-area ratios, or extended exposure times, could aid in providing a more accurate category assignment.

5. Summary

This study introduces a straightforward semi-quantitative categorization system designed to classify the corrosion behavior of metals in corrosive environments. The primary objective of the proposed system is to categorize metals based on their corrosion behavior, facilitating an improved understanding of corrosion mechanisms. This understanding, in turn, can be leveraged to design more sustainable and effective corrosion protection strategies and inhibitors [33–36].

Given the intricate interplay of material characteristics, corrosive medium properties, and corrosion process conditions, comprehensive consideration and documentation of all relevant parameters are essential when applying this categorization system. As a diverse array of systems undergo testing and classification, alongside a nuanced understanding of

the underlying physico-chemical phenomena governing each system, it is anticipated that meaningful trends and insights will emerge.

The potential wealth of knowledge that can be generated by this categorization system has the capacity to advance the understanding of corrosion phenomena, laying the groundwork for innovative corrosion inhibition strategies. This progress propels advancements in corrosion science, offering valuable tools to address corrosion-related challenges and fostering the development of more resilient materials and corrosion mitigation strategies across diverse industrial contexts. Moreover, the proposed categorization system can serve as a valuable dataset to train machine learning algorithms for classifying and predicting corrosion behavior in various environmental conditions. Such an approach has the potential to minimize time-consuming and cost-effective experimental testing, mitigating the risk of failures and catastrophes in industry.

Corrosion mitigation strategies may encompass the selection of more suitable materials for specific applications, the design of enhanced materials and alloys, and the formulation of improved corrosion inhibitor techniques. Furthermore, the classification system provides guidance for identifying the most appropriate analytical techniques for studying specific systems. Finally, the method presented herein has the potential to extend beyond the degradation of metallic materials and find applications in polymers, ceramics, and/or composites. This comprehensive approach contributes to the broader field of materials science, offering a multifaceted framework for addressing corrosion challenges and advancing the development of resilient materials.

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Conflicts of Interest: Author Francisco Malaret was employed by the company Nanomox Ltd. The author declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Appendix A

Table A1. Summary of advantages of disadvantages of common techniques used for surface characterization in corrosion studies. Based on the literature review of the scientific literature. UHV stands for ultra-high vacuum.

Technique (Acronym)	Applications	Advantages	Disadvantages and Limitations
Auger Electron Spectroscopy (AES) [20]	Elemental analysis of surface	Fast technique	• Semi-quantitative • Limited chemical information
Atomic Force Microscopy (AFM) [29]	• Corrosion studies • Surface topography analysis	• Quantitative technique for surface morphology. • Vacuum is not required. • Provides 3-D images. • Small instrument easy to operate.	• Smooth surfaces provide more accurate results.

Table A1. Cont.

Technique (Acronym)	Applications	Advantages	Disadvantages and Limitations
Digital Holographic Microscopy (DHM) [28]	Surface topography analysis	• Rapid acquisition of full-field 2D holograms. • Complete amplitude and phase information of optical field. • Sub-nanometer vertical precision with a large depth of field. • Non-contact and non-destructive imaging. • Ability to image in air and in aqueous suspensions	• Commercial instruments are not widely available and are costly. • The vertical range of imaging in a reflection mode setup is limited to the depth of focus of the objective lens. • Imaging of rough surfaces can be difficult. • The lateral resolution is limited by optical diffraction.
Grazing Incidence X-ray Diffraction (GIXRD) [26]	Surface structure determination, crystallography	• Well established and easily accessible technique. • Large database of different compounds. • Relatively easy analysis of surface long range order.	• Sample surface needs to be flat and smooth. • Analysis of ultra-thin surface layers (<10 nm) is not possible. • Power samples introduce artifacts.
High-Resolution Electron Energy Loss Spectroscopy (HREELS) [26]	Surface structure determination	• Extremely surface-sensitive with a detection limit of about 0.01% monolayer. • Analysis the mode of bonding between the adsorbate and substrate. • Detectability of a wide range of vibrational energies.	• Analysis of the mode of bonding between the adsorbate and substrate. • Only surface-adsorbed species are analysed. • Not used for identifying unknown adsorbate species. • UHV requirement.
Medium Energy Ion Scattering (MEIS) [26]	Surface structure determination	• Simultaneous measurement of composition and structure. • High sensitivity to structural parameters. • Depth profiling with high resolution.	• Quantification of atomic concentration and depth profile is difficult. • Requirement of UHV
Rutherford Backscattering Spectrometry (RBS) [20]	Depth profile measurements	• Depth profile analysis. • Rapid analysis. • Direct and simple conversion.	• High accelerator required.
Special Modulation Interferometry (SMI) [28]	Lack of electrochemical data offered by the technique	• Can be performed simultaneously with electrochemical measurements. • Offers sub-nm vertical resolution, sub-μm lateral resolution, and high speed, as well as speckle-free phase imaging using a Linnik interferometer.	Not discussed in the scientific literature

Table A1. Cont.

Technique (Acronym)	Applications	Advantages	Disadvantages and Limitations
Scanning Tunnelling Microscopy (STM) [28]	Corrosion studies, surface topography analysis	- Atomic resolution. - Surface mapping of conductive materials.	- Less preferred compared to AFM. - Tunnelling current from tip to sample surface may influence corrosion measurement.
Secondary Ion Mass Spectrometry (SIMS) [20]	Compositional profile of surface	Very high sensitivity.	- Destructive technique. - Sometimes unreliable results.
Surface Extended X-ray Absorption Fine Structure (SEXAFS) [26]	Surface structure determination	- Most sensitive to short range order bond lengths and orientations. - Surface sensitivity is like that of XPS or AES.	- Cannot identify the difference in bonding nature of an element. - Synchrotron facility required.
X-ray Absorption Spectroscopy (XAS) [20]	- XANES-oxidation state analysis. - EXAFS-radial distribution of atoms around particular atom	Any form of sample (powder, film, etc.) can be analysed; the analysis is element-specific.	- Possible damage to samples, especially in case of biological corrosion. - EXAFS cannot distinguish between atoms with very close atomic numbers such as N and O. - Coordination number analysis is conducted by the Debye–Waller factor, involving curve fitting, a possible source of error
X-ray Fluorescence (XRF)	Elemental composition analysis.	- Can analyze a wide range of elements with high sensitivity and accuracy. - Handles various sample types without extensive preparation. - Provides rapid and simultaneous analysis of multiple elements.	- Affected by matrix effects, interferences, background noise, calibration standards, and instrument performance. - Semiquantitative, as light elements are not detected.
X-ray Photoelectron Spectroscopy-Valence Band (XPS-VB) [26]	Surface phase and chemistry analysis	- Simultaneous identification of both the surface phase and chemistry in situ, especially for air-sensitive samples. - Both the phase and chemistry correspond to an identical surface area and thickness (<10 nm).	- UHV is necessary. - Poor lateral resolution (>10 µm). - Poor sensitivity due to low signal-to-noise ratio.

Table A1. Cont.

Technique (Acronym)	Applications	Advantages	Disadvantages and Limitations
Vertical scanning interferometry (VSI) [28]	Surface analysis	• Accurate ability to measure 3D surface profiles. • Capability to manage a high vertical range of height (1 mm). • With the utilization of an objective lens with magnifications from 10× to 200×, VSI has the ability to produce images with large fields of view (FOVs). • The capability to offer higher vertical resolution (1 nm to 5 nm) over large height changes (up to 1 mm). • It offers a rapid, non-contact, and non-destructive data acquisition process.	• Requires scanning along the object's vertical domain ($5 \mu\text{m s}^{-1}$ to $80 \mu\text{m s}^{-1}$). • The lateral resolution can be sub-micron ($0.5 \mu\text{m}$ to $1.2 \mu\text{m}$). • The surface measurements are often conducted ex situ in the air. • Often requires a specialized Mirau objective lens and an anti-vibration table. • VSI must perform vertical scanning to acquire its volumetric interferometric data, which means the scanning measurement could be too slow for real-time inspection.

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