
**Corrosion of metals and alloys —
Electrochemical measurement of ion
transfer resistance to characterize
the protective rust layer on
weathering steel**

*Corrosion des métaux et alliages — Mesurage électrochimique de la
résistance au transfert d'ions pour caractériser la couche de rouille
protectrice sur l'acier autopatinable*

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Weathering steel containing Cu, Ni, P, etc., is widely used for bridges, buildings, towers and other structures because of its maintenance-free characteristics resulting from the formation of a protective and adhesive rust layer when exposed to the atmosphere.

This feature makes it possible for weathering steel to be used without any surface treatment. It requires, however, several years of exposure to form the protective rust on untreated surfaces. During this period, quite complicated reactions occur within the rust layer affected by local environmental factors^{[1][2]}. Therefore, there is a strong need for a quantitative and non-destructive monitoring method for the assessment of the protective rust layer condition on weathering steel structures^[3].

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Corrosion of metals and alloys — Electrochemical measurement of ion transfer resistance to characterize the protective rust layer on weathering steel

1 Scope

This document specifies a method for the electrochemical measurement of ion transfer resistance of the rust layer formed on weathering steel alloys in order to assess their protective properties against corrosion thereafter^[3]. This method uses an electrochemical AC impedance measurement^{[4][5][6][7][8]} together with harmonic analysis, to identify the ion transfer resistance, and a rust thickness measurement to characterize the stability of the protective rust layer in terms of corrosion protection under used environments.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2178, *Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method*

ISO 9223, *Corrosion of metals and alloys — Corrosivity of atmospheres — Classification, determination and estimation*

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

ion transfer resistance

resistance against an ionic current migrated by the voltage difference through media

Note 1 to entry: In this document, “media” means the rust layer and electrolyte.

3.2

protective rust layer

layer formed on *weathering steel* (3.3) after long-time exposure to the atmosphere that enables corrosion resistance

EXAMPLE Patina on Cu alloys.

Note 1 to entry: The protective rust layer usually consists of a double-layered structure comprising a dense protective rust layer (a-Rust) underneath a crystalline FeOOH rust layer.

3.3

weathering steel

low-alloyed steel containing Cu, Ni, P, etc. that allows for a *protective rust layer* (3.2)

3.4 harmonic current

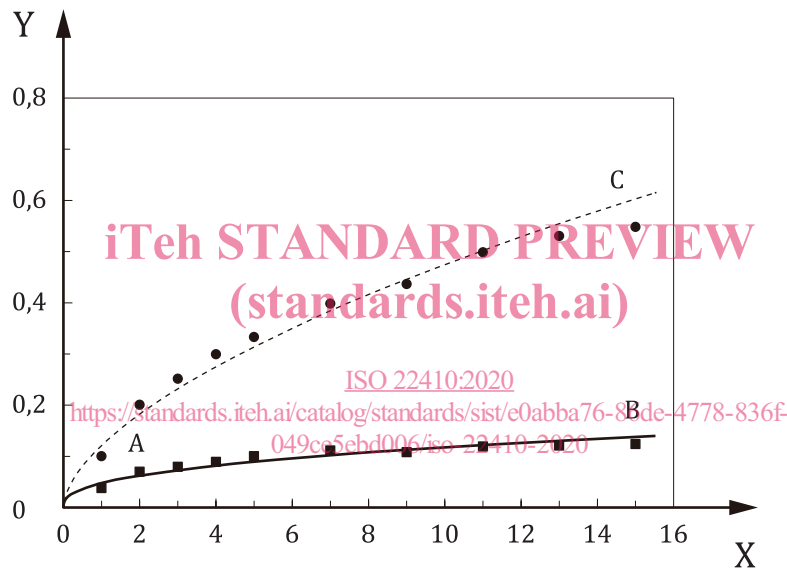
current with a frequency that is a multiple of the primary current

Note 1 to entry: A higher-order harmonic current means a nonlinear component of frequency current response under regular frequency voltage to a system.

4 Corrosion resistance by weathering steel

4.1 Formation of rust

Generally, weathering steel exhibits a good corrosion resistance after long-time exposure to the atmosphere, as shown in [Figure 1](#). This is because it forms a protective rust layer adhered to the substrate steel. However, the formation of the protective rust layer depends on the atmospheric environment and the time elapsed. Sometimes, severe corrosion can occur because there is no protective rust layer, in particular when chloride ion hinders the formation of such rust.



Key

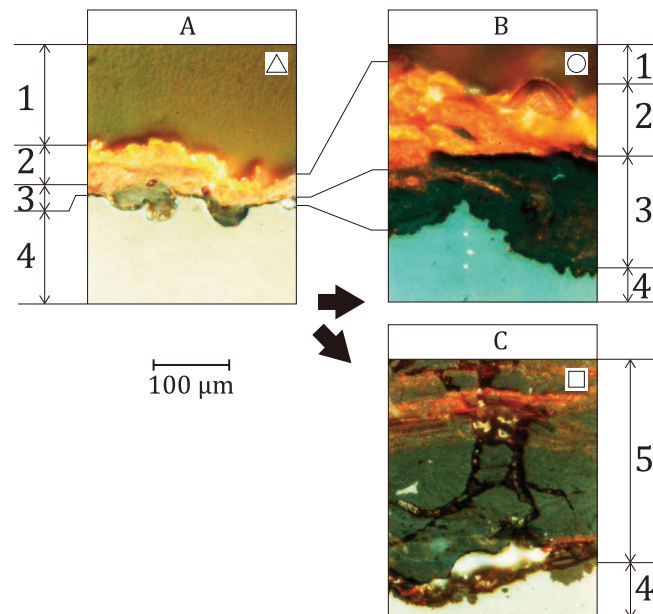
X	elapsed time, in years	A	initial rust
Y	corrosion depth, in mm	B	protective rust
		C	unprotective rust

Figure 1 — General effect of atmospheric corrosion on weathering steel over time

The protective rust usually consists of a double-layered structure, as shown in [Figure 2](#). It can be seen whether or not the rust has grown by examining a cross-sectional view of the whole rust under a microscope, as shown in [Figure 2](#). The expected features of the corrosion resistant rust are:

- a) an outer layer consisting mainly of crystalline FeOOH;
- b) an inner layer consisting mainly of amorphous or very fine crystalline iron oxyhydroxides with an enrichment of Cu, Ni, P, etc.

In initial rust and protective rust, the outer bright rust layer consists of crystalline FeOOH, whereas the inner dark rust layer has amorphous rust (very fine crystal).

**Key**

A	initial rust	1	resin
B	protective rust	2	FeOOH
C	unprotective (anomalous) rust	3	a-Rust
		4	metal (steel)
		5	thick Fe ₃ O ₄

NOTE 1 “a-Rust” means amorphous or very fine crystalline iron oxyhydroxides with an enrichment of Cu, Ni, P, etc.

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NOTE 2 “Anomalous rust” means anomalously grown rust.

Figure 2 — Classification of typical rust layers based on cross-sectional observations made using a polarized light microscope

4.2 Typical types of rust by cross-sectional view

Based on the cross-sectional microscopic observations of numerous rust layers on weathering steels exposed to various locations for various periods (see [Figure 2](#) and Reference [3]), the rust layers can be typically classified into three types: initial rust (unknown), protective rust and anomalous rust (unprotective).

- Initial rust: Islands of thin amorphous (very fine crystals) rust are visible at the steel-rust interface under a crystalline rust layer. The total thickness is usually less than 400 μm. The rust layer can be observed either at the initial stage of service or after long-term exposure to a mildly corrosive environment. The corrosion loss of the steel is very small.
- Protective rust: An amorphous or very fine crystalline rust layer covers the whole surface area under the crystalline rust layer (FeOOH). The total thickness is usually between 40 μm and 400 μm. This is the ideal protective rust layer for weathering steel.
- Anomalous rust: FeOOH exists both at the external surface and along crack surfaces. The rest of the rust consists of crystalline Fe₃O₄ (usually confirmed by X-ray analysis). The total thickness is more than 400 μm. The rust contains numerous cracks, sometimes bearing sea salt. It tends to form when the wetting period is excessive and/or accumulated salt is present.

The initial rust or protective rust can be identified by observing the cut samples under a microscope, although this type of characterization is destructive. The anomalous rust can be identified by observing a thickness measurement of rust of $> 400 \mu\text{m}$.

A non-destructive monitoring method to identify the rust layer on weathering steel is to take electrochemical measurements of the ion transfer resistance.

5 Measurement of ion transfer resistance

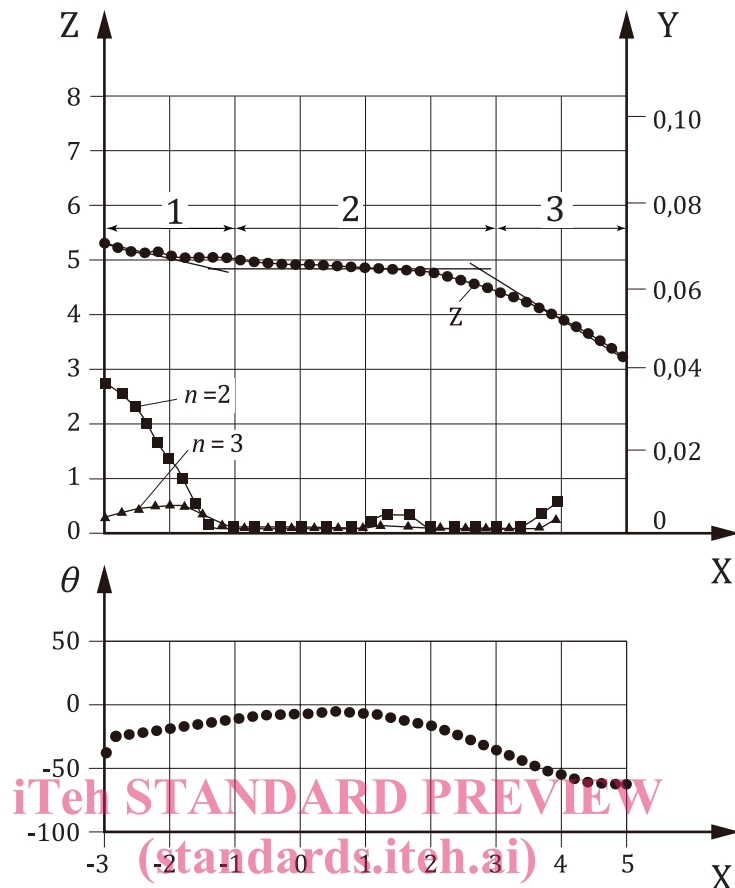
5.1 General

The protective properties of rust can be determined by measuring the ion transfer resistance through the dense and adhesive rust layer. This technique consists of using electrochemical impedance together with harmonic current measurements to identify the ion transfer resistance from the rust layer and its thickness measurement. In this case, the current is linear to potential, meaning there is almost no higher-order harmonic current. Many existing steel structures made of weathering steel under various atmospheres and periods are measured using a double-probe type of portable monitoring instrument. The thickness of the rust is measured, usually by using an electromagnetic device, and the gathered data provide a criterion map by which to characterize the property of the rust layer formed on the weathering steel, and therefore to determine whether the existing rust layer is effective as a corrosion protective barrier.

5.2 Electrochemical impedance and harmonic current measurements

In order to obtain the ion transfer resistance as a measure of the protective properties of the rust layer, the electrochemical impedance and harmonic current measurements are taken in a laboratory on various kinds of rusted low-alloyed steels exposed to the actual atmosphere over a long period. A conventional three-electrodes measurement is adopted with an Ag-AgCl reference electrode. For a standard impedance measurement, the system consists of a potentiostat and a frequency response analyser, which are controlled by a computer. An aqueous solution of 0,1 M Na_2SO_4 opened to the air is usually used as an electrolyte because the impedance parameters of the rust layer exhibit little change when this solution is used. This solution generally does not cause degradation or changes in the rust layer in a relatively short time. All the measurements are performed at room temperature (about 25°C), which simulates the measurement practice as used in the field.

A typical impedance and second and tertiary harmonic current response for a weathering steel covered with protective rust is shown in [Figure 3](#), where three distinct regions of frequency exist.

**Key**

X	frequency, in log hertz (Hz)	1	impedance region 1
Y	harmonic current, in $\times 10^{-6}$ amperes per square centimetre (A/cm ²)	2	impedance region 2
Z	impedance, in log ohms per square centimetre (Ω/cm^2)	3	impedance region 3
θ	phase shift, in degree		

NOTE $n = 2$ and $n = 3$ represent the second and the third harmonic currents, respectively.

Figure 3 — Typical impedance and the higher-order harmonic current spectra for a dense protective rust layer on weathering steel (0,1 M Na₂SO₄ solution)

Region 1 is the frequency region of less than approximately 10^{-1} Hz, where the phase shift deviates negatively from zero and the impedance is dependent on the frequency to some extent. This is called the “pseudo-capacitive impedance” since the slope of $\log Z/\log f$ is not equal to -1 , which would be exhibited in an ideal capacitor. The second and third harmonic currents are also observed, thereby indicating corrosion resistance resulting from charge transfer reactions. The higher order harmonic current is observed only when the current-potential relationship is nonlinear, e.g. the current-potential relationship depicted by the Butler-Volmer equation, on which the charge transfer reaction is based. Pseudo-capacitance in this region, of which the electrical connection should be theoretically parallel to the corrosion resistance, can be attributed to the double-layer capacitance at the steel-rust interface.

Region 2 is the frequency region between approximately 10^{-1} and 10^3 Hz, where the phase shift is nearly zero and the frequency dependence of the impedance can be almost disregarded. This is called the “pseudo-resistive impedance” since the slope of $\log Z/\log f$ is not perfectly zero. The current response in this pseudo-resistive impedance region is judged to be linear because the higher-order harmonic currents are nearly zero. Therefore, this resistance can be attributed to ion migration, which normally exhibits a linear current-potential relationship, i.e. follows Ohm’s law, through the tightly adherent inner protective rust layer. This resistance is called the “rust layer resistance” (the ion transfer resistance through the rust).