Evaluation of Anticorrosive Coatings on Carbon Steel in Marine Environments: Accelerated Corrosion Test and Field Exposure

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This study assesses the behavior of two paint systems applied to A-36 steel, commonly used to cover industrial structures in marine environments. Accelerated tests were carried out in a salt spray chamber with a maximum of 3000 hours of exposure, while other tests were conducted in the field in five areas in Chile over a period of two years. Coatings were assessed with measurements of thickness, adherence, and blistering. The behaviors of these coatings were assessed using electrochemical impedance spectroscopy (EIS techniques, measuring the evolution of an impedance module at 0.1 Hz. The results show that, after two years of exposure or after 3000 hours in the salt spray chamber, the two coatings still present adequate protective properties, with an impedance module value log |Z| greater than $10^6 \,\Omega \,\mathrm{cm}^2$. However, for all tests, comparing C5MB and C5IB coating systems, the latter is always less protective for the steel.

Keywords: Atmospheric exposure, accelerated test, corrosion, carbon steel, organic coatings.

1. INTRODUCTION

Industrial buildings built in marine environments are affected by corrosion, particularly due to saline fog that comes inland from the coastline. This fog increases the corrosion rates of carbon steel in structures [1-4], and is very relevant in Chile due to the length of its coastline [5-8].

One of the most common methods for protecting structures exposed to very aggressive atmospheres is the use of organic anti-corrosive coatings, which are an excellent option for increasing durability and protecting materials. Generally, organic coatings are more widely used due to low cost and ease of application. Such coatings are generally applied in three layers in a coating system: a first priming layer, a middle layer, and a finishing layer. The coating system acts as a barrier against aggressive media, considerably increasing material service life and leading to significant savings on operation and maintenance costs [9-12].

Technically, coatings form a barrier against the diffusion of water, oxygen, and ions through high dielectric resistance (inhibition of anodic and cathodic reactions); and through passivation of the metal with anti-corrosive pigments, i.e., some primer coats use paint that functions as cathodic protection, with particular use of Zn. The performance of a coating system, in general, is influenced by the nature of the vehicle used, the type of metal or alloy to be protected, the environment to which the material is exposed, the thickness of the paint, and the application conditions [13-14].

There are several factors that determine coating durability, including the nature, composition, and thickness of the coating; type and preparation of the substrate; and the method used to apply the paint. The paint is also affected by exposure to the environment, with factors like oxygen, humidity, temperature, environmental contaminants, solar radiation (UV and others), amount of rainfall, and wind speed and direction [15-18].

The present study assesses two coatings commonly used on carbon steel structures in Chile. These coatings were exposed to marine environments using accelerated tests in a salt spray chamber (3000 hours of exposure) and to field tests over a period of two years. The coatings differ in the primer coat (a Zn-rich epoxy vs. a self-priming epoxy) and in the top coat (epoxy vs. polyurethane).

2. MATERIALS AND METHODS

2.1. Materials

2.1.1. Metal substrate

A-36 carbon steel, measuring 10x10x0.3 cm, was used as the metallic base for applying paint. Chemical composition was determined through Optical Emission Spectrometry using a SPECTROMAXx device (Table 1).

С	Si	Mn	Р	S	Cr	Ni	Mo	Al
0.185	0.136	0.388	0.014	0.008	0.032	0.020	0.002	0.037
Cu	Co	Ti	Nb	V	W	Sn	В	Fe
0.012	0.008	0.001	< 0.001	0.002	0.009	0.012	0.0008	rest

Table 1. Chemical composition of the A-36 Steel (%).

2.1.2. Coatings

Steel samples were subjected to near-white blast cleaning (SSPC-SP10) before coating application to the steel surface (Airases Graco 56:1). Application was performed under controlled

environmental conditions, i.e., atmospheric humidity, ambient temperature, substrate temperature, and dew point, and painted samples were controlled for wet thickness, dry thickness, and porosity.

Two coatings were assessed in marine environments: S1 (C5MB) and S2 (C5IB). Their characteristics are shown in Table 2. Fig. 1 shows SEM cross-sections, obtained with a Carl Zeiss Evo MA 10 microscope.

Table	2	Coatings
I abit	4.	Coamgs.

Coating	Primer coat	Middle coat	Top coat	Total thickness (µm)
C5MB	Zn-rich Epoxy	Epoxy enamel	Polyurethane enamel	306 ± 37
C5IB	Self-priming epoxy	Epoxy enamel	Epoxy enamel	322 ± 36



Figure 1. Cross-sections, initial appearance of painted steel with different coatings (SEM). S1=C5MB S2=C5IB

2.2. Field-test procedure

The chosen test sites were five locations in Chile (Arica, Iquique, Huasco, Los Vilos, and Quintero), whose geographic coordinates are shown in Table 3, and all of which are coastal (marine atmosphere). The study was conducted over a period of 24 months (January 2014 – February 2016).

Supports made of galvanized steel were set up at each test site to hold the coated and uncoated steel samples. Samples were exposed at an angle of 45° with respect to the support, and were separated by plastic isolators, in accordance with ISO 9223 and ASTM G-50 standards [19-20].

Test site	Latitude	Longitude	Distance to the sea (m)	Height (masl)
Arica	18°28'50 S	70°19'47 W	2	4
Iquique	20°11'57 S	70°08'19 W	55	8
Huasco	28°46'53 S	71°22'22 W	88	21
Los Vilos	31°88'71 S	71°50'39 W	0	2
Quintero	32°45'08 S	71°29'07 W	5.5	2.4

Table 3. Geographic coordinates of the test stations.

2.3 Meteorological and pollution data

Weather stations were also installed at the study sites to obtain monthly data on temperature (T), relative humidity (RH), amount of rainfall, wind speed, and solar radiation. Devices were also installed to take bimonthly measurements of chloride and sulphur dioxide content in the air. The wet candle method was used to measure atmospheric chlorides. This method consists of wrapping a strip of gauze around a glass tube; the ends of the gauze are submerged in a solution of 20% glycerin in distilled water. The chlorides deposited from the air are measured by mercurimetry in the presence of diphenylcarbazone and bromophenol indicators, and the results are expressed in mg Cl⁻ m⁻² day⁻¹[21].

2.4 Corrosion testing for bare samples

The corrosion rate of bare steel was evaluated every six months by taking mass loss measurements in triplicate (ASTM G50) [20].

2.5. Evaluation of the coats

The thickness of all painted samples, with or without exposure in the salt spray chamber (Ascott, ASTM B-117) [22] or in the field, was measured in accordance with ISO 2808 using a digital Elcometer 456 [23]. Adhesion strength was measured via traction, in accordance with ASTM D4541 [23], using an Elcometer model 106; and also via abrasion, in accordance with ASTM G65. Visual evaluation of blistering was carried out with the Evans Cross technique, in accordance with ASTM-D714. Brightness was measured using a BYK Gardner micro-TRI-glass brightness meter, while color was measured using a Data color 650 TM Spectrum. The FTIR-ATRU spectra were taken using a Perkin Elmer device, model Spectrum Two.

The resistance of the coatings was measured using electrochemical impedance spectroscopy (EIS Bode spectra recorder in a frequency range between 100 kHz to 0.1 Hz at the corrosion potential.

The corrosion potential was imposed for 30 minutes before each measurement in order to ensure a stationary EIS condition). These measurements were taken every 500 hours (for the accelerated test in the salt spray chamber) and every 6 months (for the field tests) using an AUTOLAB Potentiostat / Galvanostat model 302A with EIS FRA32 module in a Na₂SO₄ 0.1 M solution at 20 \pm 2°C. A three-electrode cell was used: saturated calomel, as the reference electrode (E° = +0.242 V vs. ENH); painted samples, as the working electrode (area of exposure of 19.635 cm²); and platinum, the counter-electrode.

3. RESULTS AND DISCUSSION

3.1. Characterization of the test atmosphere

The average data obtained during the study period for the meteorological variables measured at the stations are shown in Table 4. Mean temperature is consistent with the geographical zones where the test stations are located. For example, Arica and Iquique are warmer than Huasco, Los Vilos, and Quintero. Relative humidity at stations in Huasco, Los Vilos, and Quintero is above 80%, with the highest values mainly during the first and last hours of the day (8 pm and 8 am). The average total annual rainfall varies among the five study sites, with the highest values in the central part of the country (Quintero). With regard to wind speed, Iquique has the highest values compared to the other sites. Solar radiation is slightly higher in Iquique than at the other sites, as cloud cover there is scarce, unlike the central part of the country.

Station	T (°C)	RH (%)	Accumulated rainfall (mm)	Wind speed and direction (m/s)	Accumulated solar radiation (kW/m ²)
Arica	19.8	68.6	7.7	7.8 (SW)	4126.6
Iquique	19.5	67.6	0.3	10.1 (S)	4732.1
Huasco	15.4	81.3	95.3	5.7 (SW)	4362.9
Los Vilos	12.8	86.5	318.8	8.6 (SSW)	4133.7
Quintero	13.1	84.4	734.4	7.1 (W)	3449.5

Table 4. Meteorological variable averages at different sites for 24 months.

Table 5 shows the average values for pollution agents, time of wetness (TOW), and the corrosivity category for the test stations after 24 months of exposure. TOW is determined as a function of RH and temperature, counting the number of hours in the year for which RH is greater than or equal to 80% and the temperature is above 0°C. The lowest value (τ 4) was obtained for the Arica station, located in the north of Chile. Atmospheric chloride content values were similar at all stations, classifying as S2 in most cases (60-300 mg/m²day); the only outlier was Huasco, registering a classification of S1 (3-60 mg/m²day). Atmospheric SO₂ content values were also consistent among all stations: P0 (<10 mg/m²day). Therefore, and in accordance with ISO 9223, corrosivity categories for

study sites are: between C3 and C4 in Huasco; C4, for the station in Arica; and C5 for the stations in Iquique, Los Vilos, and Quintero.

Station	SO ₂ deposits (mg/m ² day)	Chloride deposits (mg/m ² day)	TOW (%)	Corrosivity Category (ISO 9223)
Arica	2.54	65.89	56.7	$P_0S_2\tau_4 / C4$
Iquique	4.97	87.60	63.6	$P_0S_2\tau_5 \ / \ C5$
Huasco	4.76	26.56	74.5	$P_0S_1\tau_5 / C3-C4$
Los Vilos	3.99	135.44	89.5	$P_0S_2\tau_5 \ / \ C5$
Quintero	8.24	114.74	81.4	$P_0S_2\tau_5 \ / \ C5$

 Table 5. Average levels (24 months) of pollution agents and time of wetness, and corrosivity categories for test stations.

3.2. Corrosion of bare samples

Fig. 2 shows average variations in thickness loss as a function of exposure time. After 24 months of exposure to marine environments, the steel with the greatest loss was at the station in Quintero (222.2 μ m), followed by the stations in Arica (190.3 μ m), Los Vilos (187.1 μ m), and Iquique (112.7 μ m), with the lowest degree of loss of thickness seen at the station in Huasco (71.9 μ m). Fig. 2, also shows that steel loss does not level out at any of the test sites over the study period in question, with a clear linear trend at the stations in Quintero, Arica, and Los Vilos.



Figure 2. Steel thickness loss as a function of exposure time.

Fig. 3 shows average variations in steel corrosion rates at different exposure sites. The general behavior is that corrosion rate decreases over time, though the degree depends on the protective nature of the corrosion product formed on the steel and on the characteristics of the medium. In line with ISO 9226 [24], the corrosivity categories for A-36 carbon steel after a year of exposure at the different sites are as follows: Quintero (131.4 μ m year⁻¹, C5); Los Vilos (99.6 μ m year⁻¹, C5); Arica (90.5 μ m year⁻¹, C5); Iquique (76.0 μ m year⁻¹, C4); and Huasco (55.0 μ m year⁻¹, C4).



Figure 3. Average steel corrosion rates at different exposure sites.

3.3. Evaluation of coating deterioration

After 24 months of exposure at different test sites, coated samples showed no significant changes in paint layers, and thickness variations are less than the standard deviation for the measurements. No blisters were seen on the coat upon visual inspection, thus giving a classification of 10 in accordance with ASTM D714 [25] regarding the density and size of blisters. For the samples exposed in the salt spray chamber for 3000 hours, neither C5MB nor C5IB showed blistering or significant variation in the thickness of the coat.

Table 6 shows the results of adherence measurements for the two coatings, initially and after exposure for 24 months at the test sites. The data obtained confirms that during the study period, the coatings did not lose adherence to the substrate, and that any faults are due to paint layer. However, the coatings demonstrated different behaviors when subjected to accelerated salt spray tests (Table 7). The C5IB system (epoxy primer) suffers adhesion fault after 2500 hours of exposure, while the C5MB system did not show loss of adherence of the primer coat. This demonstrates the importance of the

presence of a primer with zinc particles in order to resist the presence of water once it passes the layer of paint.

		C5MB	C5IB
	Pressure, (MPa)	13.3	12.3
			Cohesion/
Initial	Type of fault/layer	Cohesion/top	between top and
condition			middle
Arica	Fault, (%)	15-35	>65%
	Pressure, (MPa)	6.3	4.5
Arico	Type of fault/layer	Adherence	Cohesion/top
Anca	Fault, (%)		0-5
	Pressure, (MPa)	7.1	4.6
Iquiquo	Type of fault/layer	Adherence	Cohesion/top
Iquique	Fault, (%)		0-5
	Pressure, (MPa)	5.5	5.0
Uuasaa	Type of fault/layer	Adherence	Cohesion/top
Truasco	Fault, (%)		5-15
	Pressure, (MPa)	5.9	3.8
Los Vilos	Type of fault/layer	Adherence	Cohesion/top
	Fault, (%)		15-35
	Pressure, (MPa)	5.8	5.8
Quintara	Type of fault/layer	Adherence	Cohesion/top
Quintero	Fault, (%)		>65%

Table 6. Coating adherence after 2 years exposure at respective test stations.

Table 7. Adherence measurements, C5MB and C5IB coatings after various salt spray exposure times.

Coating	Exposure time (hours)	Pressure (MPa)	Type of fault/ layer	Fault (%)
	0	14.2	Cohesion/ top	15-35
	1500	11.0	Cohesion/top	>65
	2000	12.7	Cohesion/top	15-35
C5MB	2500	11.9	Cohesion/ top	35-65
CJMB	3000	10.8	Cohesion/ top and middle	35-65
	0	12.3	Cohesion/top	>65
	1500	9.6	Cohesion/ top and middle	>65
C5IB	2000	6.1	Cohesion/ between middle and primer	15-35
	2500	7.2	Adhesion/primer	15-35
	3000	8.5	Adhesion/ primer	>65

Results for brightness and color measurements (expressed in GU and in CMC tolerance system units, respectively) for different samples are shown in Tables 8 and 9. Loss of brightness is mainly caused by solar radiation - for those samples exposed only in the salt spray chamber, loss of brightness is lower in comparison with painted samples exposed to the environment. Samples exposed at the station in Iquique show greater variation in brightness and color due to the effects of solar radiation, as this site has the highest level of accumulated solar radiation (4732.1 KWm⁻²). The comparison of the two coatings shows that the top coat of polyurethane has a higher degree of brightness overall, and less loss of brightness, over 24 months of exposure. Results for color measurements and visual inspections showed that the green epoxy paint saw higher degrees of color loss.

	Brightness, (GU)				Color, (CMC)	
Station	C5MB		C5IB		24 months	
	initial	24 months	initial	24 months	C5MB	C5IB
Arica		48.8		8.4	0.63	1.30
Iquique		42.4		1.0	0.36	2.15
Huasco		43.9		1.7	0.86	1.63
Los Vilos	72.4	53.9	13.9	1.3	0.55	1.81
Quintero		47.1		1.2	0.85	1.67

Table 8. Brightness and color measurements for coatings after 2 years exposure at test stations.

Table 9. Brightness and color measurements for C5MB and C5IB after salt spray exposure.

Salt spray chamber	Bright	ness, (GU)	Color, (CMC)
	initial	3000 hours	Color difference
C5MB	72.4	64.9	0.30
C5IB	13.9	10.4	2.30

Fig. 4 shows the FTIR-ATRU spectra for the top polyurethane coat (C5MB) on the painted samples before and after exposure in Arica and Los Vilos (C5 corrosivity category, similar accumulated solar radiation [Tables 4, 5]), and after exposure in the salt spray chamber in the accelerated test. The % reflectance in different bands is decreased for samples exposed to the atmosphere; though less, it also decreases for salt spray chamber samples when compared to the initial samples without exposure. A decrease in % reflectance of the band at 3377.5 cm⁻¹ indicates a break in the secondary amine bonds. The bands at 2930.2 cm⁻¹ and 2861.0 cm⁻¹ indicate a decrease in the acyclic C-H bond. The decrease in the bands at 1726.6 cm⁻¹ and 1687.4 cm⁻¹ is attributed to a change in the carbonyl of the amide, suggesting degradation of the polyurethane groups. The decrease in the bands at 1452.2 cm⁻¹ and 1240.2 cm⁻¹ suggests a reduction in the C-H and C-O bonds.



Figure 4. FTIR-ATRU spectra of C5MB sample exposed for 24 months, Arica and Los Vilos (A), salt spray chamber (B).

Fig. 5 shows that a decrease in % reflectance is also seen for the samples painted with a top coat of epoxy resin, both for samples exposed to the atmosphere (in Los Vilos and Arica) and samples exposed in the salt spray chamber (again, when compared to the initial samples before exposure). The decrease in the band at 1013.8 cm⁻¹ indicates a break of the ether functional group bonds, which is the main group present in an epoxy resin. The decrease in the band at 1235.0 cm⁻¹ suggests a reduction in the C-O bond due to degradation of the resin.

The FTIR-ATR spectra demonstrate evident degradation of the polyurethane and epoxy resins, whether exposed in climate conditions (UV radiation) or in controlled conditions in a salt spray

chamber (Cl⁻ ions). Comparatively between the two resins, the epoxy shows a greater decrease in its characteristic bands, indicating a greater degree of degradation.



Figure 5. FTIR-ATRU spectra of C5IB sample exposed for 24 months, Arica and Los Vilos (A), salt spray chamber (B).

Fig. 6 shows abrasion measurement results for coated samples at different stations. Loss of thickness is notably higher for C5MB, with the biggest difference over the same period observed at the Arica station. All else equal, abrasion is higher in materials of lower hardness.



Figure 6. C5IB and C5MB thickness loss from abrasion after 24 months exposure at test stations.

As shown in Fig. 7, and irrespective of atmosphere, C5MB samples (polyurethane top coat) presented significantly better behavior. There is very little blistering, and only negligible corrosion, which may be ascribed to the presence of Zn in the primer. For the C5IB samples (epoxy resin primer), there is damage on the cut, and corrosion products and blistering. This is very evident in particularly more aggressive atmospheres, such as at the stations in Arica, Iquique, Los Vilos, and Quintero.

Fig. 8 shows the surface appearance of samples after exposure for 3000 hours in a salt spray chamber. This test corroborates the weaker behavior of the C5IB coating with epoxy primer versus the C5MB coating with Zn-rich epoxy primer coat; the latter acts synergistically as barrier and sacrificial anode.



Figure 7. Surface appearances of C5MB and C5IB samples after 24 months of exposure.



Figure 8. Surface appearance of (a) C5MB and (b) C5IB samples after 3000 hours of exposure in a salt spray chamber.

Characteristics of both types of coating were also analyzed via electrochemical impedance spectroscopy (EIS) based on |Z| values obtained from Bode spectra recorded over frequency interval 100 kHz to 0.1 Hz. Measuring |Z| values at 0.1 Hz is a commonly employed method in the literature for evaluating protective coating behaviors [9, 25]. In this sense, a coating offers excellent protection if log |Z| is > 10, good protection if log |Z| is > 8 and poor protection if log |Z| is ≤ 4 .

Figures 9 and 10 show the behavior of average |Z| values measured at 0.1 Hz for both coatings exposed at the field stations. The same figures also show changes in |Z| values obtained for up to 3000 h in the salt spray chamber for both coating samples. Initially, high |Z| values found for both coatings show typical capacitive responses, indicating undamaged paint. However, both coatings show gradual decreases of |Z| with increased exposure times, probably due to water diffusing into paint film, and thus loss of protection to the metallic base substrate due to coating degradation (see Table 10); this pattern is more apparent in the C5IB system. Indeed, this aspect is further confirmation of the FT-IR spectra analysis, where the top layer of this paint system showed important degradation both in field samples (regardless of test site location) and under controlled conditions in the salt spray chamber.

It is necessary to highlight that this behavior is also greater for samples exposed in areas with higher corrosivity categories, such as Los Vilos and Quintero (C5). However, all painted samples exposed at different stations over 2 years show good protective behavior in an aggressive medium when considering the impedance module values obtained (log |Z| > 8) [9, 25].

EIS coating behavior includes accelerated salt spray chamber tests, to serve as analog to field site marine atmospheres (see figures 9 and 10). Chamber results show the same trends as does the field data; the C5IB system shows greater |Z| decreases at 3000 exposure hours. However, impedance module values are lower for the samples exposed in the chamber than for samples exposed in the field. This was to be expected, due to the aggressive nature of the atmosphere inside the salt spray chamber (temperature of 37 °C, 100% humidity, and 3.5% chloride solution) and due to the ultrafine electrolyte particles that can easily penetrate the microstructure and deposit on coating surfaces, and thus deteriorate them. In spite of the above, the impedance modules value (*ac.* 6 < log |Z| < 7) at 3000 hours of exposure, indicate that coatings maintain their protective effect for the steel. Lee *et al.* [9] have shown that the degree of surface deterioration of a paint layer can be associated with the real electrical resistance of this layer (R_p).



Figure 9. Variation in impedance module values for C5MB at test stations and salt spray chamber.



Figure 10. Variation in impedance module values for C5IB at test stations and salt spray chamber.

This value can be obtained through simulation from experimental Bode spectra, though can be adequately correlated with $|Z|_{0.1\text{Hz}}$ values, where R_p corresponds to the most important barrier layer. In our case, this approach was correct for both field samples and the salt spray chamber. Table 10 shows

 $|Z|_{0.1Hz}$ values for each coating from initial condition to after two years of field exposure. The same table also shows the value of $|Z|_{0.1Hz}$ obtained in the salt spray chamber obtained after 3000 hours of exposure.

Table 10. Variation in impedance module values ($|Z| / \Omega \text{ cm}^2$) for both coatings after 2 years (test sites) and 3000 hours (salt spray chamber) of exposure.

Coating	Initial	Arica	Iquique	Huasco	Los Vilos	Quintero	Salt Spray Chamber
C5MB	$1.7 \ge 10^{10}$	3.7 x 10 ⁹	$2.0 \ge 10^9$	3.8 x 10 ⁹	1.1 x 10 ⁹	9.3 x 10 ⁸	$6.2 \ge 10^7$
C5IB	$3.6 \ge 10^{10}$	$2.0 \ge 10^8$	3.4×10^8	9.0 x 10 ⁸	2.7×10^8	1.7 x 10 ⁸	1.6 x 10 ⁶

As can be observed, the values summarized in Table 10 show that both coatings suffer slight surface deterioration, regardless of installation location. Indeed, $|Z|_{0.1\text{Hz}}$ values, with respect to initial conditions, only decrease by two orders of magnitude. Based on these results, after two years of environmental exposure, both coatings continue to provide adequate protection for A-36 carbon steel. On the other hand, on the basis of the results obtained in the salt spray chamber, and although the same trends can be found, it is not possible to determine correlations; the effect of UV radiation is not considered in the accelerated test. This factor directly influences coat deterioration, as was shown by Petry *et al.* [26] when comparing paint in the field and in a salt spray chamber that did include the variables of UV radiation and ozone content.

4. CONCLUSIONS

In this study, atmospheric corrosivity classifications were C5 for tested field stations in Arica, Los Vilos, and Quintero, and C4 in Huasco and Iquique.

The steel samples coated using C5MB and C5IB presented no significant changes or blistering during exposure time, either in the field or in the salt spray chamber. Surface color variation in samples painted with C5IB (a green epoxy enamel layer) showed a higher degree of color run and loss of adherence at 2500 hours of exposure in the salt spray chamber.

The Evans Cross test showed weaker behavior for C5IB, both in the field and in the salt spray chamber. The primer for this system is a self-priming epoxy, in comparison to the Zn-rich epoxy used in the C5MB coating.

Observations are adequately supported by EIS measurements. Deterioration of coated samples exposed to the environment and installed in the salt spray chamber were monitored using an impedance module value at 0.1 Hz, which found that, in spite of 2 years of exposure in the field or 3000 hours in the salt spray chamber, coatings continue to provide an adequate protective barrier, with impedance module values log |Z| greater than $10^6 \Omega \text{ cm}^2$. However, in all tests used to compare C5MB and C5IB coatings, the latter consistently showed weaker protective behavior for the steel.

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