

# Effect of Surface Contaminants Remained on the Blasted Surface on Epoxy Coating Performance and Corrosion Resistance

† Kwang Ki Baek, Chung Seo Park, Ki Hong Kim,  
Mong Kyu Chung, and Jin Hwan Park<sup>1</sup>

H970, R&D Center, Hyundai Heavy Industries, Co., Ltd.  
1 Jeonha-Dong, Ulsan, KOREA, 682-792

<sup>1</sup>Dept. of Industrial Chemical Eng., Pukyong Nat'l Univ., Busan, Korea 608-737

One of the critical issues in the coating specification is the allowable limit of surface contaminant(s) - such as soluble salt(s), grit dust, and rust - after grit blasting. Yet, there is no universally accepted data supporting the relationship between the long-term coating performance and the amount of various surface contaminants allowed after grit blasting. In this study, it was attempted to prepare epoxy coatings applied on grit-blasted steel substrate dosed with controlled amount of surface contaminants - such as soluble salt(s), grit dust, and rust. Then, coating samples were subjected to 4,200 hours of cyclic test(NORSOK M-501), which were then evaluated in terms of resistance to rust creepage, blistering, chalking, rusting, cracking and adhesion strength. Additional investigations on the possible damage at the paint/steel interface were carried out using an Electrochemical Impedance Spectroscopy(EIS) and observations of under-film-corrosion. Test results suggested that the current industrial specifications were well matched with the allowable degree of rust, whereas the allowable amount of soluble salt and grit dust after grit blasting showed a certain deviation from the specifications currently employed for fabrication of marine vessels and offshore facilities.

**Keywords** : soluble salt, rust, grit dust, blasting, protective coating, Sa 2.5

## 1. Introduction

For successful application of high-performance epoxy protective coatings, each shipyard has established its own practice and inspection to assure a quality work for hull structures and outfittings. One of the main issues for coating application is the standards for surface preparation, mostly based on ISO Standard(such as, ISO 8501-1). Normal surface preparation requires the surface, free from visible oil, grease and dirt and from mill scale, rust, and other foreign matters. Either blast cleaning or hand/power tool cleaning is used. For the secondary surface preparation at the block stage, the additional requirements, such as removal of fume of welding and flame cutting, spatters in the way of welding beads are specified.<sup>1)</sup>

Removal of surface contaminants, however, such as soluble salt(s), grit dust, and rust is not clearly specified in terms of their allowable limits, and generally these were based on the coating manufacturer's recommendation. The

presence of soluble salts, such as chlorides and sulfates at the coating/steel interface would incur and/or accelerate the osmotic blistering of organic coating as well as the under-film-corrosion of steel substrate. They also cause general degradation of coating performance, such as poor adhesion, cathodic disbondment, rust creepage, in a rather short time.<sup>2)</sup> The rust or grit dust would diminish the paint's wettability, and work as obstacles for bonding formation.<sup>3)</sup>

Therefore, several international organizations, such as NACE, ISO, NORSOK have established standards regarding the types and allowable amount of surface contaminants. Establishment of these standards for surface preparation for coating could be a formidable task considering wide variety of coatings and their thickness not to mention of their service environments, which could be dry/wet, hot/cold, fresh/salt water, and so on.

Among the contaminants mentioned above, the most controversial one is the soluble salt(s), even though the other two are also discussed for their threshold limit for quality performance of protective coatings, especially for the immersion service. So far, many technical references

† Corresponding author: mat@hhi.co.kr

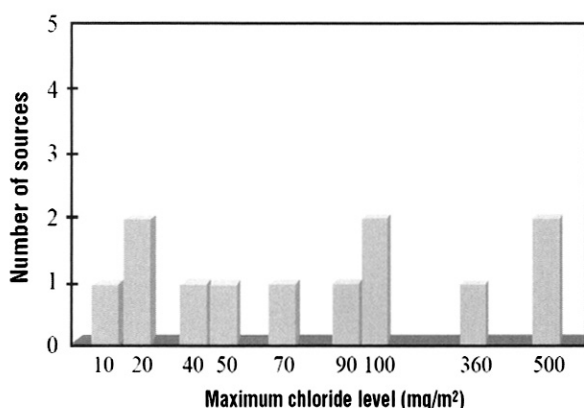


Fig. 1. Max. allowable chloride concentration<sup>1)</sup>

have been reported on this issue, and some of the results were adopted in the relevant specifications and standards as a recommendation for surface preparation methods for marine vessel and offshore facilities. There are, however, no systematic test results showing a definite answer to satisfy both the owners and the builders for several reasons. One of the obstacles to obtain proper technical data is the difficulty of preparing coating specimens with systematically controlled amount of a specific contaminant. Another one, which has been neglected in a certain degree, is the difficulty in selection of the accelerated corrosion test methods to predict the long term performance of modern epoxy coatings used for immersion service.

Over 30 technical papers were published and 18 authors based on 11 independent investigations proposed maximum allowable levels of chloride contamination. In those studies, various coating materials and corrosion tests were employed. The experimental data of these studies, as graphically represented in Fig. 1,<sup>2)</sup> showed that in most cases a maximum allowable chloride level of 20 to 100 mg/m<sup>2</sup>, has been reported.

Whether these limits are adequate criteria or not, however, is yet to be further explored due to the fact that there is no universally accepted data revealing the relationship between the long-term coatings performance and the amount of various surface contaminants allowed after grit blasting. Thus, in this study, the efforts were focused on to determine the thresholds for these contaminants employing the coating specimens prepared on grit-blasted steel substrate dosed with controlled amount of surface contaminants.

## 2. Experimental method

Steel panels (10 cmx15 cm) were blasted to Sa2.5 finish with 70  $\mu$ m anchor profile. All blasted steel surfaces were

Table 1. Coating specimen preparation

Steel panel	CR mild steel(100 x 150 x 5 mm)
Surface preparation	Edge grinding, Alkali degreasing, Sa2.5 Gr. (profile $70 \pm 5 \mu$ m)
Coating material	Resin: bisphenol A, Hardener: polyamide, Pigment: Talc, TiO <sub>2</sub> , Al S.V.R. = 80%
D.F.T.	2 coats, $260 \pm 10 \mu$ m, Autogun airless spray

degreased with acetone and methanol for removing of oil, grease, or other contaminants before systematic application of each contaminant. Two coats of commercial, modified epoxy(avg. 260  $\mu$ m, Dry Film Thickness, D.F.T.) coatings were applied, as summarized in Table 1, via an auto air spray gun on the contaminated panels with controlled amount of surface contaminants(soluble chloride salt, rust and grit dust). The selected coating materials was commercial epoxy coatings currently widely used in ship building industries for Water Ballast Tank(W.B.T.) sections and other immersion service.

### 2.1 Soluble salt

Calculated amount of NaCl was added to 10 ml distilled water, which was diluted with methanol to make 200 ml solution. Then, 0.8 ml of the chloride added methanol was carefully spread on the grit blasted steel surface(150 cm<sup>2</sup> area) by using a micropipet. Then, specimens were immediately dried by a hot air blower to prevent flash rust development.

Soluble chloride salt content on the blasted steel was prepared to be 9 different grades having concentration of 0, 10, 30, 50, 70, 100, 150, 200, 400 mg/m<sup>2</sup>. The type of salt was confirmed by a Inductively Coupled Plasma (ICP) measurement, and salt concentration of each specimen was measured and confirmed by using an ion chromatography and the boiling extraction method.

### 2.2 Rust

The blasted steel panels were prepared following the same method used for salt contaminated coating samples. These panels were put in a chamber, of which environment was maintained at 50 °C and 70–80% Relative Humidity. After spraying water on the steel panels with a portable atomizer, rust development was monitored based on ASTM D610 standard. Fig. 2 and Fig. 3 show the rust status of each specimen(Gr.9–Gr.1) prior to coating application at the low and high magnification, respectively. Considering the actual coating practice of avoiding coating application on the heavily rusted surface, the most severely rusted condition of Gr.1 and Gr.2 were eliminated for the

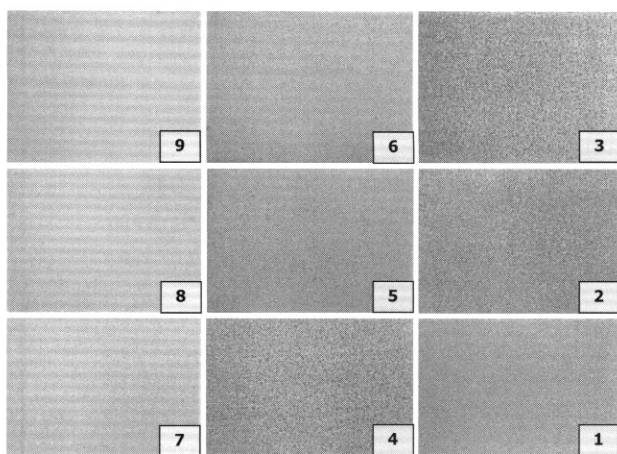


Fig. 2. Developed rust and grade based on ASTM D610(low mag.)

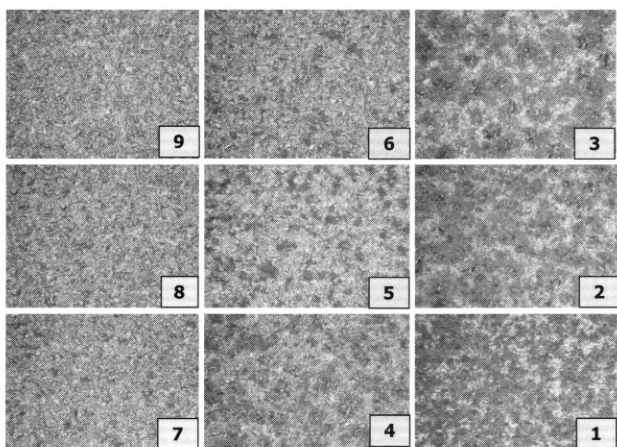


Fig. 3. Developed rust and grade based on ASTM D610(x100 mag.)

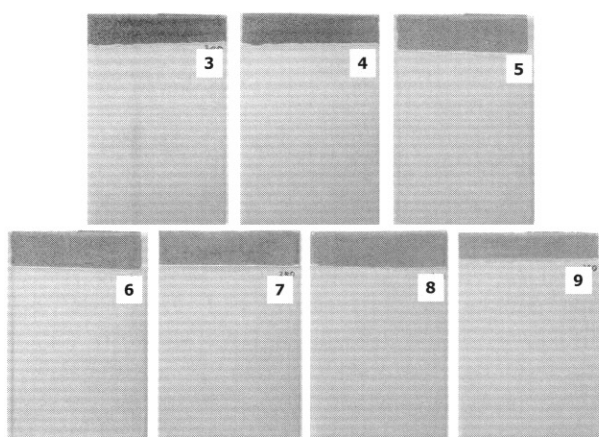


Fig. 4. Rust specimens after application of coating

further tests, and other rusted specimens after application of epoxy coating are shown in Fig. 4.

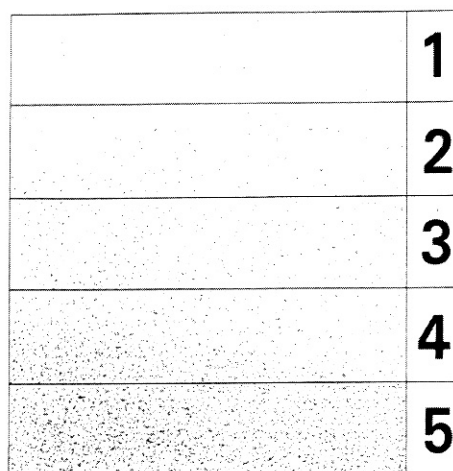


Fig. 5. Grit dust reference from ISO 8502-3

### 2.3 Grit dust

The blasted steel panels were prepared following the same method used for salt contaminated coating samples. To select proper grit dust contaminant representing an actual coating practice, grits from a block coating shop in a ship yard were collected, sieved with a 100  $\mu\text{m}$  grid sieve. These grit dusts were manually applied on the steel panels, of which dust grade were determined by using replica tape method based on ISO 8502-3 standard, clause 6.3(quantity class), as shown in Fig. 5.

### 2.4 Corrosion test and evaluation

Corrosion resistances of the surface contaminated coating samples were evaluated by using, the NORSOK M-501(Revision 4) cyclic tests. Among several accelerated corrosion test methods for coatings, the NORSOK M-501 (Revision 4) cyclic test was far more severe than others, which was composed of three, separated steps, which were 80 hours of exposure to QUV weathering chamber condition, 72 hours of salt spray chamber exposure, followed by 16 hours of drying in an ambient environment. Fig. 6 shows the test sequence with coating specimens mounted

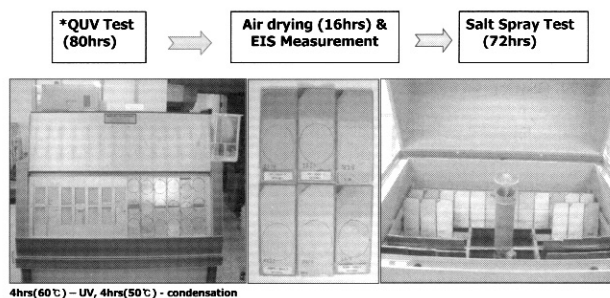


Fig. 6. NORSOK M-501 cyclic test methods(168hrs for a single cycle)

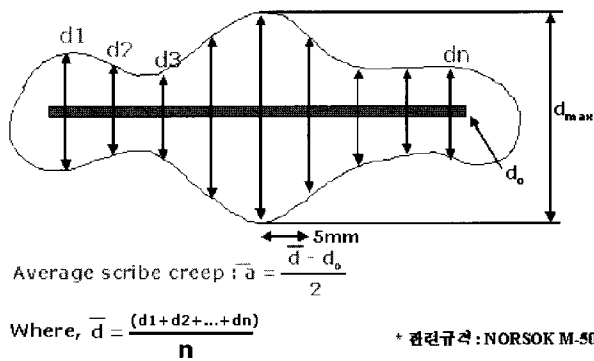


Fig. 7. Evaluation of corrosion creepage

Table 2. Acceptance criteria after NORSOK M-501 Test

Test Item	Acceptance Criteria
Corrosion Creepage	less than 3 mm
Blistering(ISO 4682-2)	Rating 0 (no blister)
Chalking(ISO 4628-6)	Max. rating 2
Rusting(ISO 4628-3)	Rating 0 (rust area 0%)
Cracking(ISO 4628-4)	Rating 0 (x10 mag.)
Adhesion(ISO 4624)	Min 5.0 MPa

on the testing equipment. All coatings were subjected to 25 cycles, which took a total of 4,200 hours. After finishing the total cycles, several evaluations were carried out, as summarized in Table 2, and the acceptance criteria for the evaluation were also listed in the Table. Rust creepages of coating were measured following the NORSOK M-501 standard, as shown in Fig. 7.

To evaluate the coatings barrier properties, Electrochemical Impedance Spectroscopy (EIS) was used to measure the AC Impedance of coating during the drying stage of each cyclic test. For EIS measurement, each coating specimen was dried for 8 hours, and immersed in 0.5 M NaCl solution for 30 minutes before initiation of impedance measurement at 10 kHz frequency.

### 3. Results and discussion

#### 3.1 Soluble salt

Corrosion creepage measured from the chloride contaminated coatings after 25 cycles were less than 3 mm for the coatings applied on the steel surfaces having chloride concentration of 0~70 mg/m<sup>2</sup>, whereas those of the coatings applied on the steel surfaces having chloride concentration of 100~400 mg/m<sup>2</sup> were over 3 mm. The corrosion creepage of coatings after the 25 cycles of corrosion test are shown in Fig. 8. For evaluation of corrosion creepage, NORSOK M-501 measurement method was

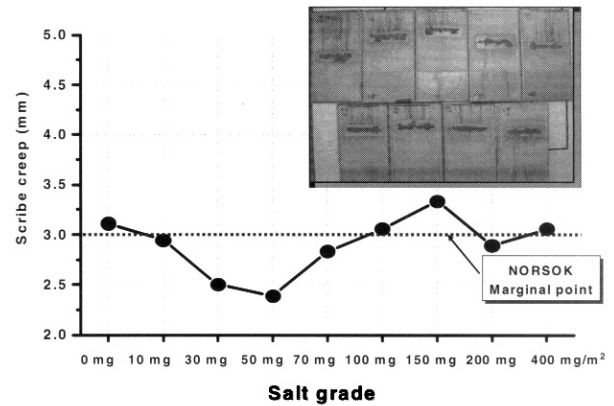


Fig. 8. Corrosion creepage vs. chloride concentration

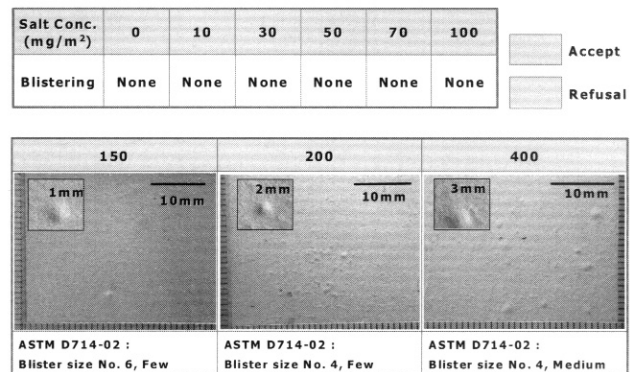


Fig. 9. Blistering vs. chloride concentration

used.

Blistering was not observed for the coatings applied on the steel surfaces having chloride concentration of 0~100 mg/m<sup>2</sup>, whereas the coating applied on the steel surfaces of 150 mg/m<sup>2</sup> and 200 mg/m<sup>2</sup> chloride showed blistering degree of No. 6 and No. 4, respectively, which could be categorized as the "few" blistering, or "one or a few blisters per unit area" condition as in the ASTM D714-02. The coating having the largest chloride concentration of 400 mg/m<sup>2</sup> showed blistering degree of No. 4, which was "medium" blistering, or "several blisters per unit area" condition, as shown in Fig. 9.

EIS measurements on the coatings with up to 400 mg/m<sup>2</sup> of surface chloride contamination showed the AC impedance in the range of  $1 \times 10^7 \sim 2 \times 10^8$  ohm/cm<sup>2</sup> at 10kHz up to 25 cycles of corrosion tests, as shown in Fig. 10. Based on the other study on the EIS measurement of organic, protective coating, in which it was suggested that impedance value lower than  $10^6$  ohm/cm<sup>2</sup> at 10 kHz was the indication of coating film's degradation,<sup>4)</sup> these results indicated that there was no distinguishable degradation or reduction of barrier property of coating due to the salt contamination of the surface.

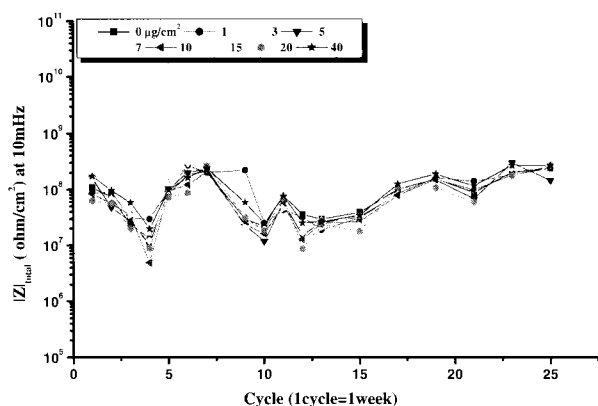


Fig. 10. AC impedance variation vs. chloride concentration

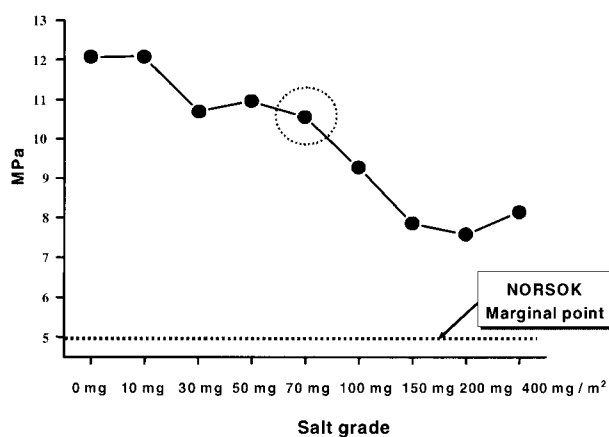


Fig. 11. Adhesion strength vs. chloride concentration

The adhesion strength of all coatings with up to 400 mg/m<sup>2</sup> of surface chloride contamination were measured larger than the minimum requirement of 5 MPa after 25 cycles, as shown in Fig. 11. It showed a general trend that the increase of salt concentration at the steel surface would cause the decrease of the adhesion strength of the coating due to the under-film-corrosion.<sup>4),5)</sup> On the other hand, for all coatings with up to 400 mg/m<sup>2</sup> of surface chloride contamination, no chalking, surface rusting and cracking were observed at the coating surface upon completion of the entire cycles.

It is known that the presence of soluble salt on the steel surface would promote moisture diffusion into the coating/steel interfacial region, which resulted in under-film-corrosion,<sup>4),5)</sup> and the absence of surface corrosion in protective coatings does not guarantee that there is no under-film-corrosion. Thus, coated films of all the specimens were separated from the steel substrate after completion of evaluations. For this purpose, the coating specimens were immersed in a thinner solution at 80 °C for 48 hours, and dried, which caused spontaneous peeling of the coated film, revealing the steel surface to facilitate

direct examination of under-film-corrosion. The results, as shown in Fig. 9, revealed that the under-film-corrosion was not observed for the steel surfaces of chloride concentration of 0~70 mg/m<sup>2</sup>, whereas the steel having 100 mg/m<sup>2</sup> chloride experienced 3% of under-film-corrosion, in the pinpoint rust form of "5-P" condition. For the steel with chloride concentration of 150 mg/m<sup>2</sup> and 200 mg/m<sup>2</sup> showed 10%(4-G, in general rusting form) and 16%(3-G, in general rusting form) of the under-film-corrosion, respectively. Finally, the steel with 400 mg/m<sup>2</sup> chloride showed 33% of the under film corrosion, in the general rust form of "2-G" condition.

Based on the above test results, the maximum allowable chloride level was suggested to be 70 mg/m<sup>2</sup> for immersion service. However, the maximum allowable chloride level may vary with different coating systems and in different service condition.

### 3.2 Rust

Corrosion creepage measured from the rust contaminated coatings after 25 cycles was less than 3 mm for the coatings applied on the steel surfaces having rusts of 9~3 grade, whereas it was noticeable that slight increase of creepage was observed with the increase of rust. For evaluation of corrosion creepage, NORSOK M-501 measurement method was used.

Blistering, chalking, surface rusting and cracking were not observed at the coating surface upon completion of the test for the coatings applied on the rusted steel surfaces having rusts of 9~3 grade. EIS measurements on the rust contaminated coatings showed that the AC impedance was in the range of  $5 \times 10^7 \sim 2 \times 10^8$  ohm/cm<sup>2</sup> at 10 kHz after 25 cycles of corrosion tests, suggesting that there was no degradation of coating's barrier property in spite of rust presence at the steel surface.

The adhesion strengths of all coatings with presence of rust at the coating/steel interface were measured to be larger than the minimum requirement of 5 MPa, as shown in Fig. 12. It showed a general trend that the increase

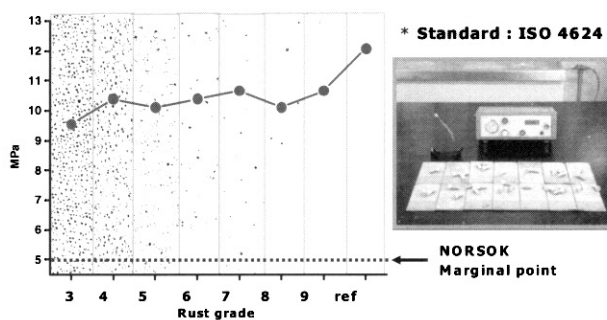


Fig. 12. Adhesion strength vs. rust grade

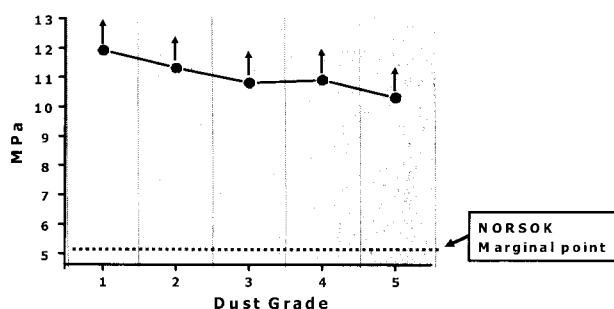


Fig. 13. Adhesion Strength vs. dust grade

of rust at the steel surface would cause the decrease of the adhesion strength of the coating, because the presence of rust would reduce the wettability.<sup>4)</sup> Recently, there are several new commercial coatings available, which can tolerate a certain surface irregularities including minor rusts on the surface. This suggests that the acceptance level of rust contamination is particularly dependent on the formulation of coating materials.

### 3.3 Grit dust

The adhesion strength of all coatings with up to grit dust Gr. 5 (quantity class), of surface contamination were measured to be over 10 MPa, larger than the minimum requirement of 5 MPa, as shown in Fig. 13. Blistering, chalking, surface rusting and cracking were not observed on the coatings with grit dust grade of 1~5. EIS measurements on the coatings with up to grit dust grade of 5 showed the AC impedance in the range of  $\sim 10^8$  ohm/cm<sup>2</sup> at 10kHz after 25 cycles of corrosion tests, suggesting that there was no degradation of coating's barrier property in spite of dust presence at the steel surface.

For cargo oil tankers coating application, the maximum allowable dust on the surface to be coated varied according to different specifications, especially for the W.B.T. section. For example, one specification proposed the ISO 8502-3 Gr.1 as the standard surface condition,<sup>6)</sup> whereas another proposal for the steel surface cleanliness before application of coating was the condition of "dust shall be removed".<sup>7)</sup> In reality, however, it is hardly possible to achieve the ISO 8502-3 Gr.1 cleanliness for the block assemblies having complex structure with numerous

vertical/horizontal members in a practical manner. Moreover, the epoxy-based coating used for ships was known for its excellent wettability, and thus, complete wetting of fine dusts on the steel surface could be anticipated. Consequently, the initial dust grade needs not be so stringent at the start of actual coating. These results implicated that grit dust of Gr.5 can be accepted as a proper surface preparation to quality coating performance.

## 4. Conclusions

Results suggested that the current industrial specifications were well matched with the allowable degree of rust, whereas the allowable amount of soluble salt and grit dust on the blasted surface showed a certain deviation from the specifications currently employed for fabrication of marine vessels. For commercial epoxy coatings generally used for ships, such as W.B.T. sections, maximum allowable chloride of 70 mg/m<sup>2</sup> and ISO 8502-3 dust Gr.5 were proposed as a required surface cleanliness.

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## References

1. H. H. I., *Painting & Inspection Practice*, Shipbuilding Division, Ulsan, Korea (2002).
2. B. P. Alblas and A. M. van Londen, *Jr. Protective Coatings Europe*, pp. 18~25 (1997).
3. B. S. Fultz and J. W. Peart, *Jr. Protective Coatings & Linings*, pp. 28~34 (1996).
4. B. R. Appleman, *ibid*, pp. 68~82 (1987).
5. G. C. Soltz, NSRP, Report 0329, U.S.A (1991).
6. TSCF, *Guidelines for ballast tank coating system and surface preparation*, England, (2002).
7. DNV, Notes No.33.1 Corrosion prevention of tanks and holds.