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EFFECTS OF CHEMICAL CLEANING PROCEDURE FOR REMOVAL CORROSION PRODUCTS TO DISSOLUTION RATE

BY
KORRAKOCH MEECHUMNARN

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(Dr.Santhad Rojanasoonthon)

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THAILAND INSTITUTE OF SCIENTIFIC AND TECHNOLOGICAL RESEARCH

RESEARCH PROJECT NO. 32-07 RESEARCH AND DEVELOPMENT OF ANTI-CORROSION MATERIALS

REPORT NO. 1

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ศึกษาผลของสารเคมีที่ใช้ล้าง CORROSION PRODUCTS บนผิวโลหะต่ออัตราการละลาย

โดย กรกช มีชำนาญ

บทคัดย่อ

ได้ทำการศึกษาเกี่ยวกับสารเคมีที่ใช้ล้าง corrosion products บนผิวโลทะ ซึ่งในการ ทดลองนี้คือ common carbon steel และ cold rolled steel. ตามมาตรฐาน ISO TC/WG 156 ได้แนะนำให้ใช้ diammonium citrate solution. นอกจากนี้ยังได้ศึกษาถึง ปัจจัยอื่น ๆ ที่มีผลต่อ dissolution rate อีกด้วย.

ผลการทศลองพบว่าปัจจัยอื่น ๆ ที่มีผลต่อ dissolution rate เมื่อใช้ diammonium citrate solution ได้แก่ อุณทภูมิของสารละลาย, specimen holder และสารละลายที่ใช้ แล้ว.

EFFECTS OF CHEMICAL CLEANING PROCEDURE FOR REMOVAL CORROSION PRODUCTS TO DISSOLUTION RATE

By Korrakoch Meechumnarn*

ABSTRACT

Study was conducted on chemical cleaning solution to remove corrosion products on metal surfaces. In this experiment common carbon steel and cold rolled steel were used. According to ISO TC/WG 156 the diammonium citrate solution was recommended to remove corrosion products on steel surface. Study on factors which had effected the dissolution rate was also conducted.

From the experimental results, factors which had effected the dissolution rate when using diammonium citrate solution were solution temperature, specimen holder and used solution.

^{*}Metal and Material Technology Department, Thailand Institute of Scientific and Technological Research

1. INTRODUCTION

In atmospheric corrosion study, it is important to determine the corrosion rate of various kinds of metals which are exposed outdoor. Thus weight of metals before and after exposure should be measured, that is, the experimental data should be obtained by dissolving corrosion products without any attack on the base metal. Such dissolution procedures have been studied and specified in some international standards, such as ISO⁺ and JIS. However, no procedure has been found which does not attack the base metal absolutely. So the mass loss rate of base metal by the dissolution of corrosion products must be known since the details such as dissolution rate have not yet been disclosed or published.

Based on previous studies, it is easy to dissolve corrosion products uniformly for steel specimens exposed in urban or rural area. But steel specimens which were exposed in splash zones of coastal area were locally attacked and thickness or depth of these corrosion products varied with localized surface of the specimen. Longer time is required to remove the corrosion products completely. Therefore, larger amount of base metal is dissolved simultaneously.

In National Research Institute of Metals (NRIM), experimental works have been carried out in these past months to choose suitable methods of corrosion products dissolution from the procedures specified by ISO TC/WG 156. On evaluating these procedures, twelve

⁺ISO/IDP 8407.2 1988.

methods were chosen from the view point of waste water safety and laboratory air pollution problem, thus cyanide and concentrated hydrochloric acid were no longer employed in the study. From the results, it was found that base metals were much corroded by the three kinds of ISO procedures. The proportion of metal loss to the amount of removed rust was more than 22.0%; therefore it was recognized that the chemicals employed in the acidic mixture of these ISO procedures were not suitable for rust removal. On the other hand, the nine procedures were considered suitable since the proportion of metal loss to the amount of removed rust were 0.5-12.8%.

Hereafter, the modified procedure of iron rust removal, such as the use of electrochemical process of acid inhibitor which possesses stronger inhibiting power of bare steel dissolution should be studied.

For years, NRIM has employed sulfuric acid combined with a commercial acid inhibitor for exposed iron and steel and has obtained satisfactory results. But, instead of using commercial acid inhibitors, it is better to use other chemical substances which are suitable for inhibition. Among chemical substances recommended by ISO to be used for specimens exposed at urban and rural areas, there is doubt that using these solutions will dissolve a large amount of base metals.

Atmospheric corrosion experts in Japan and China have requested NRIM for detailed information on these rust removing procedures, chemical substances, inhibitors and operating conditions including the mass loss rate of base metal caused by these chemicals.

In the present study, the factors affecting the dissolution rate of steel specimens in diammonium citrate solution were investigated.

2. EXPERIMENTATION

2.1 Dissolution and mass loss rates

2.1.1 Solution preparation. 400 g diammonium hydrogen citrate with distilled water to make 2,000 ml (200g/l).

2.1.2 Specimen preparation

- (1) Common carbon steel being exposed for one year at various sites in Japan (Tokyo, Okinawa and Chikura) were weighed and then cleaned by using mechanical method.
- (2) Cold rolled steel was polished with emery paper No. 400 and then degreased with acetone.

2.1.3 Removal of corrosion products

- (1) Weigh the specimen.
- (2) Heat the diammonium citrate solution to the temperature of 80 $^{\frac{1}{2}}$ $^{\circ}$ C.
- (3) Put the specimen into the solution about 5 min.
- (4) Bring the specimen out.
- (5) Put the specimen into the water.
- (6) Brush the specimen with steel brush.
- (7) Rinse the specimen with distilled water.
- (8) Dry and weigh the specimen.
- (9) Do (1) to (8) every 5 min.

2.1.4 Calculation

- (1) Plot graph between mass loss and cleaning time.
- (2) Extrapolate the graph to y-axis, the mass loss of the sample was known.
- (3) Calculation of mass loss rate:

 mass loss rate = ______ mass loss (mg)

surface area (cm²) x 365 (days)

(4) Calculation of dissolution rate:

dissolution rate = mass loss (mg)

surface area (cm²) x cleaning time (min)

2.2 Factors affecting dissolution rate

In this experiment only four factors were studied as follows:

- (1) Solution temperature.
- (2) Specimen holder.
- (3) The used solution.
- (4) Solution containing NaCl.
- 2.2.1 Solution temperature. Common carbon steel and cold rolled steel specimens were cleaned in the diammonium citrate solution at temperature of 80 $^{+}$ 1°C and 100 $^{+}$ 1°C and compared the dissolution rate.
- 2.2.2 Specimen holder. Tantalum wire was used as specimen holder. Cold rolled steel specimen was cleaned in the diammonium citrate solution at $80 \, \frac{1}{2} \, 1^{\circ}$ C and compared dissolution rate both of using and not using specimen holder.
- 2.2.3 The used solution. Cold rolled steel specimen was cleaned in the used solution which was used many times to remove

corrosion products of specimens at $80 ^+1^{\circ}$ C. The dissolution rate was compared with that of the specimen which was cleaned in the new solution at the same solution temperature.

2.2.4 Solution containing NaCl. 1.0, 5.0, 10.0, 20.0, and 50.0 g/l of NaCl was added in the diammonium citrate solution. Cold rolled steel specimen was cleaned in these solution with varied amount of NaCl and then compared the dissolution rate with that of the specimen which was cleaned in the solution with no NaCl at the same temperature of 80 $^+$ 1°C.

3. RESULTS

3.1 Dissolution and mass loss rates

3.1.1 Common carbon steel. Common carbon steel being exposed at various sites in Japan: Tokyo, Okinawa and Chikura were cleaned by using diammonium citrate solution at solution temperature of 80^{+} 1° C. The results of mass loss, dissolution rate of common carbon steel at various sites were shown in Tables 1-3 and the relation between mass loss and cleaning time was illustrated in Figures 1-3.

From the results, the average dissolution rate of common carbon steel at Tokyo site was 0.138 mg/cm^2 .min; at Okinawa site was 0.145 mg/cm^2 .min; and at Chikura site was 0.213 mg/cm^2 .min.

TABLE 1. MASS LOSS AND DISSOLUTION RATE OF COMMON CARBON STEEL EXPOSED AT TOKYO SITE AND CLEANING TIME

		Sample 1		Sample 2
Cleaning time (min)	Mass loss	Dissolution rate (mg/cm ² .min)	Mass loss	Dissolution rate (mg/cm ² .min)
5	_			-
10			-	-
15	-	-	4.66	0.986
20	5.20	0.825	6.21	0.984
25	6,31	0.705	6.74	0.336
30	6.94	0.400	7.20	0.292
35	7.08	0.089	7.42	0.140
40	7.34	0.140	7.65	0.165
45	7.53	0.121	7.85	0.127
50	7.72	0.121	8.07	0.121
55	7.97	0.165	8.27	0.127
60	8.22	0 - 159	8.46	0.121
	a	vg. 0.141	avç	g. 0,134

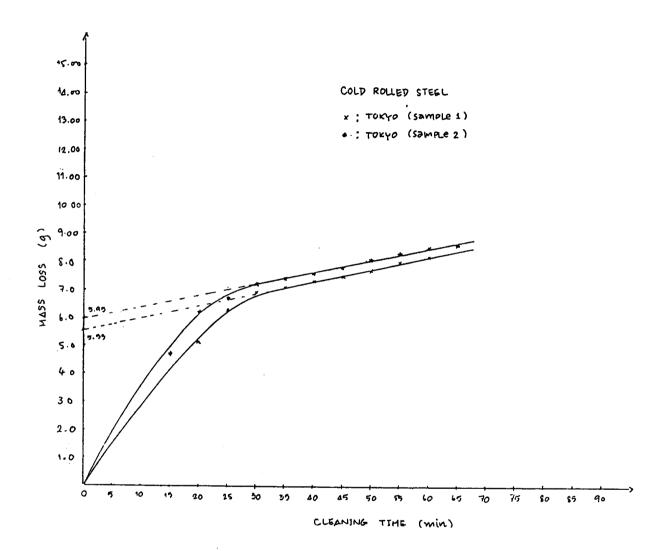


Figure 1. The relation between mass loss and cleaning time of common carbon steel exposed at Tokyo site.

TABLE 2. MASS LOSS AND DISSOLUTION RATE OF COMMON CARBON STEEL EXPOSED AT OKINAWA SITE AND CLEANING TIME

	Samp	ole 1	Samç	ole 2
time (min)	Mass loss	Dissolution rate (mg/cm ² .min)	Mass loss (g)	Dissolution rate (mg/cm² .min)
5	~	-	-	-
10	-		-	-
15	-	-	4.33	0.916
20	9.88	1.568	6.48	1.365
25	11.75	1,187	8.37	0.959
30	12.45	0,444	9.87	0.952
35	12.66	0.133	10.56	0.438
40	12.91	0.140	10.90	0.216
45	13.09	0.114	11.18	0.178
50	13.28	0.121	11.42	0.152
55	13.55	0.165	11.62	0.127
60	13.81	0 - 165	11.84	0.140
	avç	J. 0.141	av	g. 0.149

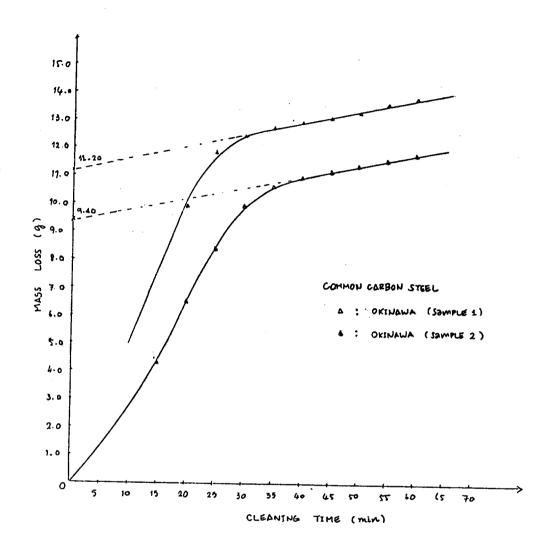


Figure 2. The relation between mass loss and cleaning time of common carbon steel exposed at Okinawa site.

TABLE 3. MASS LOSS AND DISSOLUTION RATE OF COMMON CARBON STEEL EXPOSED AT CHIKURA SITE AND CLEANING TIME

	S	ample 1	S	Sample 2
Cleaning				and the second s
time	Mass loss	Dissolution rate	Mass loss	Dissolution rate
(min)	(g)	(mg/cm ² .min)	(g)	(mg/cm ² .min)
5		-	-	-
10	-	-	-	-
15	~	-	18,24	3.860
20	16.64	2.641	19.84	1.016
25	18,95	1.467	21.14	0.825
30	20.81	1,181	22.40	0.800
35	23.11	1.460	23.54	0.724
40	24.02	0.533	24.40	0.546
45	24.54	0.356	25.07	0.425
50	24.95	0.260	25.65	0.368
55	25.33	0.235	26.02	0.235
60	25.61	0.178	26.34	0.190
65	25.86	0-159	26.74	0.241
70	26.24	0.222	27.09	0.222
75	26.58	0.216	27.41	0.203
80	26-89	0.197	27.73	0.203
	avg	0.141	avg	. 0.149

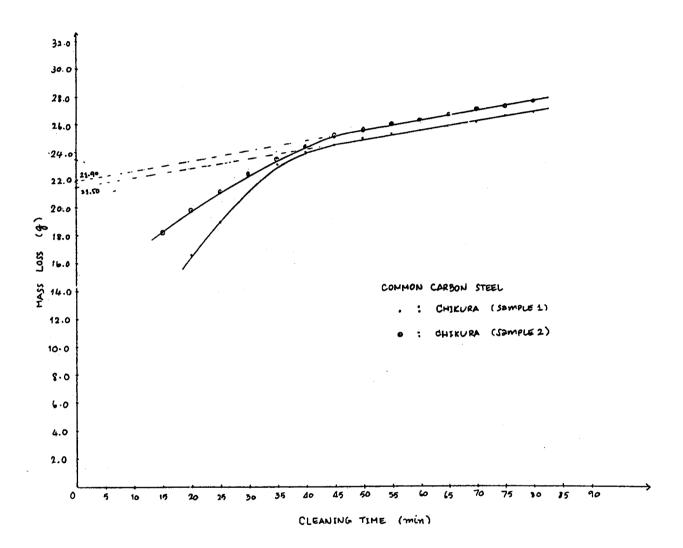


Figure 3. The relation between mass loss and cleaning time of common carbon steel exposed at Chikura site.

From Figures 1-3, the graphs were extrapolated to the y-axis, so it showed that the mass loss of samples and the surface area of the specimen was 315 cm². The mass loss rate of the samples could be calculated and the results were shown in Table 4.

TABLE 4. MASS LOSS RATE OF COMMON CAREON STEEL EXPOSED AT VARIOUS SITES IN JAPAN

	Mass loss rate (mdd)		
Exposed sites	Sample 1	Sample 2	Average
Tokyo (suburb)	4.82	5.17	5.00
Okinawa (seaside)	9.74	8.17	8.96
(50-100 m from coast)			
Chikura (seaside)	18.69	19.04	18.86
(5-10 m from coast)			

3.1.2 <u>Cold rolled steel</u>. Cold rolled steel being exposed at NRIM were cleaned by using diammonium citrate solution at solution temperature of 80 $^{+}$ 1°C. The results of mass loss, dissolution rate and cleaning time were shown in Table 5. The relation between mass loss and cleaning time was illustrated in Figure 4. From Table 5 the average dissolution rate of cold rolled steel was 0.0145 mg/cm 2 .min.

TABLE 5. MASS LOSS AND DISSOLUTION RATE OF COLD ROLLED STEEL EXPOSED AT NRIM AND CLEANING TIME

Cleaning time (min)	Mass loss (g)	Dissolution rate (mg/cm ² .min)
5	0.0456	0.0590
10	0.0752	0.0380
15	0.1021	0,0350
20	0.1235	0.0280
25	0.1405	0.0220
30	0.1582	0.0230
35	0.1704	0.0170
40	0.1826	0.0158
45	0.1944	0.0152
50	0.2053	0.0143
55	0.2188	0.0175
60	0.2308	0.0155
65	0.2412	0.0136
70	0.2536	0.0160
75	0.2626	0.0118
80	0.2734	0.0140
85	0,2810	0.0097
90	0.2928	0.0169
95	0.3038	0.0143
100	0.3168	0.0169
105	0.3289	0.0156
110	0 • 3372	0.0108

avg. 0.0145

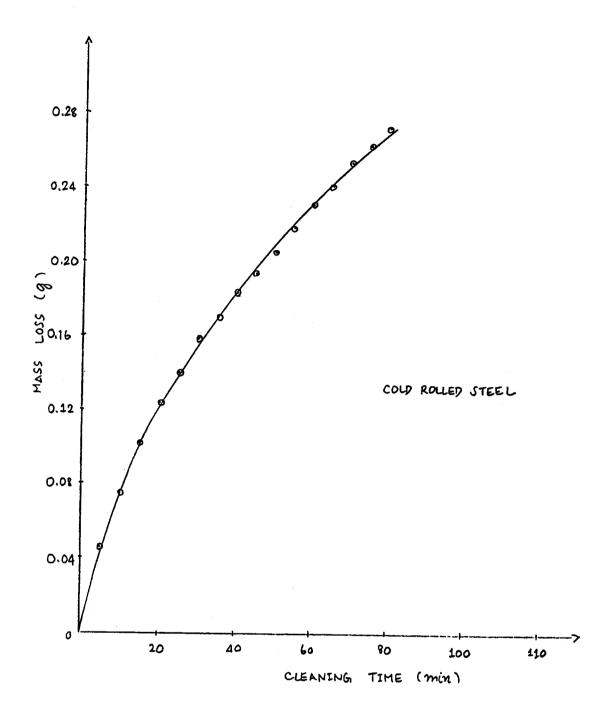


Figure 4. The relation between mass loss and cleaning time of cold rolled steel specimen exposed at NRIM.

3.1.3 Comparison of common carbon steel and cold rolled steel. Cold rolled steel and common carbon steel specimens being exposed at NRIM and Tokyo site respectively were cleaned in the same conditions by using diammonium citrate solution at solution temperature of 80 [†].

1°C. In this experiment the comparison of the dissolution rate of both specimens was illustrated. The results of mass loss, dissolution rate and cleaning time were shown in Table 6. From Table 6 it was found that the dissolution rate of common carbon steel exposed at Tokyo site was very similar to that of cold rolled steel exposed at NRIM.

TABLE 6. COMPARISON OF MASS LOSS AND DISSOLUTION RATE BETWEEN COLD ROLLED STEEL AND COMMON CARBON STEEL

	Cold	rolled steel	Common o	carbon steel
Cleaning time (min)	Mas s loss	Dissolution rate (mg/cm ² .min)	Mass loss	Dissolution rate (mg/cm ² .min)
5	7.65	0.165	0.1052	0.136
10	7.85	0.127	0.2006	0.124
15	8.07	0.121	0.2944	0.123
20	8.27	0.127	0.3842	0.117
25	8.46	0.121	0.4866	0.133
30	8.65	0.121	0.5776	0.118
	av	g. 0.130	avg.	0.125

3.2 Factors affecting the dissolution rate

3.2.1 Solution temperature. From this experiment it was found that solution temperature had effected the dissolution rate of common carbon steel and cold rolled steel. When the solution temperature increased from $80 \, \frac{1}{2} \, 1^{\circ}$ C to $100 \, \frac{1}{2} \, 1^{\circ}$ C, the dissolution rate also increased. The results were shown in Tables 7 and 8.

TABLE 7. EFFECT OF SOLUTION TEMPERATURE TO THE DISSOLUTION RATE OF COMMON CARBON STEEL

Cleaning time	Mass loss	Dissolution rate
(min)	(g)	(mg/cm ² .min)
5	~	_
10		-
15	4.66	0.986
20	6.21	0.984
25	6.74	0.336
30	7.20	0.292
35	7.42	0.140
40	7.65	0.165
45	7.85	0.127
50	8.07	0.121
55	8.27	0.127
60	8.46	0.121
65	8.65	0.121
70*	9.32	. -
75 [*]	9.66	0.216
80*	9.96	0.190

^{*} The solution temperature was 100 - 1°C.

TABLE 8. EFFECT OF SOLUTION TEMPERATURE TO THE DISSOLUTION RATE OF COLD ROLLED STEEL

Cleaning time (min)	Mass loss (g)	Dissolution rate (mg/cm ² .min)
5	0.1052	0.136
10	0.2006	0.124
15	0.2944	0.123
20	0.3842	0.117
25	0.4866	0.133
30	0.5776	0.118
35*	1.1022	-
40*	1.3875	0.370
45*	1.5834	0.254

^{*} The solution temperature was 100 - 10°C.

3.2.2 Specimen holder. In this experiment the cold rolled steel specimen which was cleaned for 110 min from 3.1.2. was used. The specimen was cleaned again at the solution temperature of $80^{\frac{1}{2}}$ 1° C and tantalum wire was used as the specimen holder. It was found that tantalum wire had little effect to the dissolution rate of cold rolled steel, as shown in Table 9. The relation between mass loss and cleaning time was illustrated in Figure 5.

TABLE 9. EFFECT OF TANTALUM WIRE HOLDER TO THE DISSOLUTION RATE OF COLD ROLLED STEEL

Cleaning time (min)	Mass loss (g)	Dissolution rate (mg/cm² .min)
115	0.3494	0.0165
120	0.3598	0.0134
125	0.3706	0.0140
130	0.3816	0.0144
135	0.3923	0.0138

Not using tantalum wire, dissolution rate average 0.0124

140*	0.4057	0.0200
145*	0.4217	0.0207
150*	0.4356	0.0162
155*	0.4479	0.0172
160*	0.4595	0.0151

Using tantalum wire, dissolution rate average 0.0178

^{*}using tantalum wire holder.

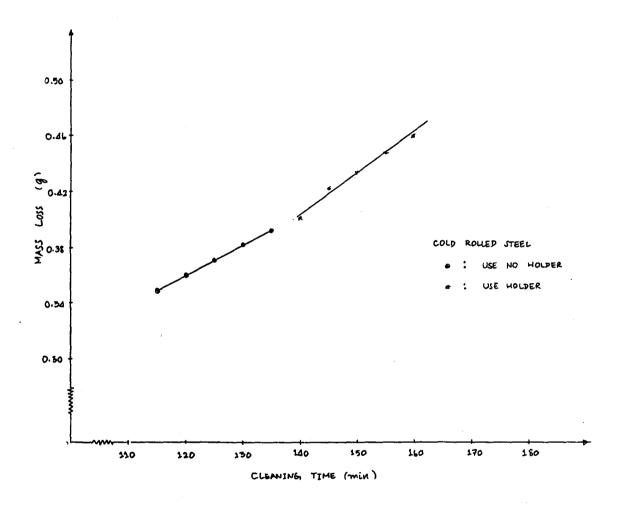


Figure 5. The relation between mass loss and cleaning time of cold rolled steel when using tantalum wire and not using tantalum wire.

3.2.3 The used solution. In this experiment it was found that at the solution temperature of $80 \pm 1^{\circ}$ C, dissolution rate of cold rolled steel in the used solution was higher than that in the new solution. The results shown in Table 10 and Figure 6 illustrated the relation between mass loss and cleaning time in this case.

TABLE 10. EFFECT OF THE USED SOLUTION TO THE DISSOLUTION RATE

Cleaning time (min)	Mass loss (g)	Dissclution rate (mg/cm ² .min)
5	0.1712	0.222
10	0.3503	0.232
15	0.5072	0.204
20	0,6744	0.217
25	0.8330	0.206
30	1,0074	0.226
35	1.1766	0.220
40	1.3350	0.206
45	1.4807	0.189
50*	1.5458	0.051
55 [*]	1.5688	0.030
60 [*]	1.5929	0.031
65 [*]	1.6222	0.038
70 [*]	1.6513	0.038
75 [*]	1.6769	0.033
80	1.9002	0.291
85	2.1039	0.264
90	2 • 3230	0 - 284
95	2.5430	0.298

^{*}Using the new solution.

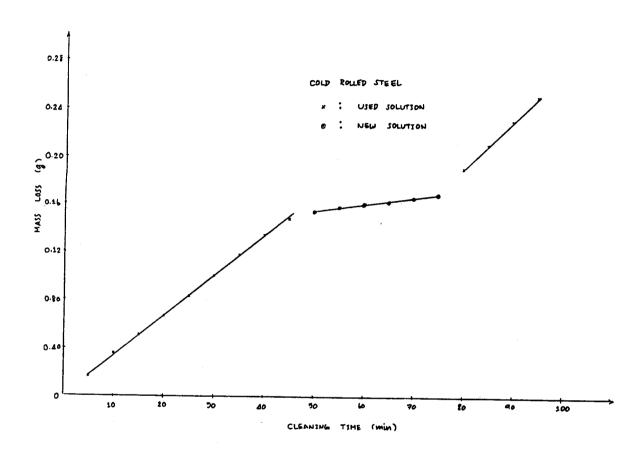


Figure 6. The relation between mass loss and cleaning time when using the new solution and the used solution.

3.2.4 <u>solution containing NaCl</u>. Because the high dissolution rate in the used solution may be the effect from NaCl, so the test was performed by adding NaCl into the solution 1.0, 5.0, 10.0, 20.0 and 50.0 g. In this experiment, it was found that NaCl had no effect to the dissolution rate of cold rolled steel. The results were shown in Table 11 (a-e).

TABLE 11. THE EFFECT OF NACL ADDED IN THE SOLUTION TO THE
DISSOLUTION RATE OF COLD ROLLED STEEL: (A) 1.0 G
(B) 5.0 G (C) 10.0 G (D) 20.0 G (E) 50.0 G

(a) NaCl added 1.0 g

	Solution		Solution + NaCl	
Cleaning time (min)	Mass loss (g)	Dissolution rate (mg/cm ² .min)	Mass loss	Dissolution rate (mg/cm ² .min)
5	0.0456	0.059	0.0386	0.0476
10	0.0752	0.038	0.0712	0.0423
15	0.1021	0.035	0.0100	0.0373
20	0.1235	0.028	0.1262	0.0343
25	0.1405	0.022	0.1474	0.0212
30	0 • 1582	0.023	0 - 1626	0.0200
	avg. 0.0342		avg.	0.0338

(b) NaCl added 5.0 g

	Solution		Solution + NaCl	
cleaning time (min)	Mass loss	Dissolution rate (mg/cm².min)	Mass loss	Dissolution rate (mg/cm² .min)
35 40 45 50 55 60	0.1704 0.1826 0.1944 0.2053 0.2188 0.2308	0.0174 0.0158 0.0152 0.0143 0.0175 0.0155	0.1757 0.1881 0.2001 0.2136 0.2281 0.2396	0.0193 0.0161 0.0155 0.0193 0.0190 0.0148
		avg. 0.0160		avg. 0.0173

(c) NaCl added 10.0 g

	Solution		Solution + NaCl	
cleaning time (min)	Mass loss	Dissolution rate (mg/cm² .min)	Mass loss	Dissolution rate (mg/cm ² .min)
65	0.2412	0.0136	0.2540	0.0199
70	0.2536	0.0160	0.2668	0.0197
75	0 - 2626	0.0118	0.2790	0.0158
80	0-2734	0-0140	0.2898	0.0134
85	0-2810	0.0097	0.3002	0.0134
	avg。0.0130		avg. 0.0164	

(d) NaCl added 20.0 g

	Solution		Solution + NaCl	
Cleaning time (min)	Mass loss	Dissolution rate (mg/cm ² .min)	Mass loss (g)	Dissolution rate (mg/cm ² .min)
90 95 100 105 110	0.2928 0.3038 0.3168 0.3289 0.3372	0.0169 0.0143 0.0169 0.0156 0.0108	0.3100 0.3226 0.3334 0.3440 0.3542	0.0158 0.0136 0.0141 0.0137 0.0132
	avg. 0.0149		avg. 0.0141	

(e) NaCl added 50.0 g

	Solution		Solution + NaCl	
Cleaning time (min)	Mass loss	Dissolution rate (mg/cm² .min)	Mass loss	Dissolution rate (mg/cm² .min)
115 120 125 130 135	0.3494 0.3598 0.3706 0.3816 0.3923	0.0165 0.0134 0.0140 0.0144 0.0138	0.3658 0.3776 0.3894 0.3999 0.4100	0.0169 0.0140 0.0154 0.0136 0.0132
	avg. 0.0124		avo	g. 0.0146

4. DISCUSSION

In this experiment common carbon steel specimens being exposed for one year at various sites in Japan and cold rolled steel specimens being exposed at NRIM were dissolved in diammonium citrate solution. Diammonium citrate was the chemical removal recommended in ISO/TC 156 to be used with corrosion products of iron and steel. It was found that dissolution rate of common carbon steel which exposed at site in Tokyo to that of cold rolled steel was very similar. Mass loss rate of common steel specimens which exposed in seaside area was higher than in suburb area. And from this experiment the factors which had some effects to the dissolution rate were solution temperature, specimen holder and the used solution. It was found that dissolution rate was high when the solution temperature was high.

For the specimen holder, tantalum wire was used as holder in this experiment. It was found that dissolution rate was slightly high when using the holder.

The last factor which had effected the dissolution rate was the used solution. It was found that dissolution rate was very high when using the used solution compared with the dissolution rate in the new solution. At first it was assumed that this occurrence might be the effect of NaCl when dissolving in the used solution from specimens, so the test was made by adding NaCl in the new solution. It was found that NaCl had no effect to the dissolution rate. Therefore, it should have more studies in the cause of high dissolution rate in the used solution.

5. CONCLUSIONS

- 5.1 Dissolution rate of common carbon steel and cold rolled steel being exposed at the same atmosphere is very similar.
- 5.2 Mass loss rate of common carbon steel specimen from seaside area is higher than from suburb area.
 - 5.3 Solution temperature has effect to the dissolution rate.
 - 5.4 Specimen holder has little effect to the dissolution rate.
- 5.5 Dissolution rate in the used solution is higher than that in the new solution.

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