

# A Study on the Effect of Corrosion Resistance According to the Composition Variety of C, Cr, N in Duplex Stainless Steel

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Recently the alloy development of duplex stainless steel has been done. On this study we studied the effect of the corrosion resistance according to the composition variety of C, Cr, N in the alloy elements of duplex stainless steel. materials which have below 0.1[mm/year] corrosion rate enable to use for corrosion-resisting materials, generally. On this experiment we inspected the effect of the composition variety of C, Cr, N in duplex stainless steel and the heat treatment, which the condition was the water quenching after the heat treatment for 1hr. The experiment was done on the basis of the ASTM G48A test, Critical pitting temperature(CPT), and ASTM G-61(Electrochemical tests for cyclic polarization).

**Keywords :** duplex stainless steel, carbon, chromium, nitrogen, corrosion rate

## 1. Introduction

Stainless Steel that are used generally are very strong about corrosive ions such as chloride ion so on, but in severe environment contained comparative high corrosive ions come from easily several corrosion such as pitting corrosion and crevice corrosion.

As we are shown a Schaeffero diagram(Fig. 1)<sup>1)</sup>, duplex stainless steel has two phase which is austenite phase and ferrite phase, and after in 1930's this stainless steel was discovered for the first time. Stainless steel coexisting autenite/ferrite two phase is called duplex stainless steel.

Each phase merits of the alloy are embossed because of coexisting two phase, and new stainless steel improved a shortcoming has developed.

Corrosion resistance and working of duplex stainless steel are very good. Duplex stainless steel contains molybdenum(Mo) for increasing corrosion resistance in sea environment, and this material resist pitting corrosion, crevice corrosion and SCC by chloride ion very well, and specially it has a good SCC resistance and is known that it is not more sensitive than austenite stainless steel.<sup>2)-5)</sup>

Microstructure ferrite phase of duplex stainless steel is known that it tends to be corroded selectively in acid atmosphere of reaction environment and sometimes to increasing corrosion rate because of being generated galvanic phenomenon with austenite phase and becoming worse.

Stainless steel of the materials which are generally

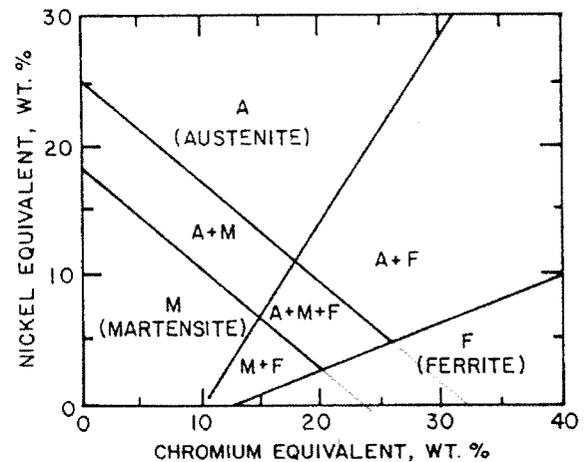


Fig. 1. Schaeffler diagram including modifications by Schneider

happened several shapes and kinds of corrosion is decided by not only alloy element but also many kinds of factors.<sup>6),7)</sup> As a among other stainless steel, duplex stainless steel is a alloy using metallography of these material. Because of being decided by a various physical, chemical character such as microstructure of the alloy, crystal particle size, particle distribution uniformity, segregation of metallic chemical compound, crevice existing in a surface geometric characteristic, existing defect and so on. For estimating a corrosion resistance, a choice of a proper test method and a construction according to test is very important.<sup>8)</sup>

Duplex stainless steel alloy elements for the corrosion resistance are chromium(Cr), molybdenum(Mo), nitrogen

(N), Tungsten(W) and so on. Chromium and molybdenum of these elements are known that they improve the corrosion resistance and we have studied a lot.

Factors resisting corrosion phenomenon in stainless steel are a fine passivation film, a high strength, a austenite structure, a low carbon content, and so on. Especially the forming of a strong passivation film improved resisting not only pitting or crevice corrosion but also stress corrosion cracking using the content of chromium and molybdenum. We can show quantitatively how strong the forming of the passivation film are.

PREW which Hiroshi Okamoto suggests he use 30 for a coefficient of Nitrogen and a half of molybdenum for a coefficient of tungsten is following.<sup>9)</sup>

$$\text{PREW} = \text{wt}\% \text{Cr} + 3.3(\text{wt}\% \text{Mo} + 1/2 \text{wt}\% \text{W}) + 30 \text{wt}\% \text{N}$$

Then if these pitting resistance numerical index equation is high, the corrosion resistance is very good in one phase of stainless steel, but in duplex stainless steel that doesn't happen all the time. As shown Fig. 2, the corrosion resistance valuation which is made by critical pitting temperature according to PREW value variety in the alloy containing tungsten is shown, and if super duplex stainless steel which PREW value is more than 50 is not developed the corrosion resistance according to increasing PREW value. As result, in one phase stainless steel standard for estimating the corrosion resistance can use PREW value, but in duplex stainless steel that is not enable to use PREW value.

Therefore Garner<sup>10)</sup> made an empirical formula( $\% \text{Cr} + 3.3\% \text{Mo}$ ) called Cr-Equivalent. This is gotten by estimating corrosion rate in  $\text{FeCl}_3$  solution according to

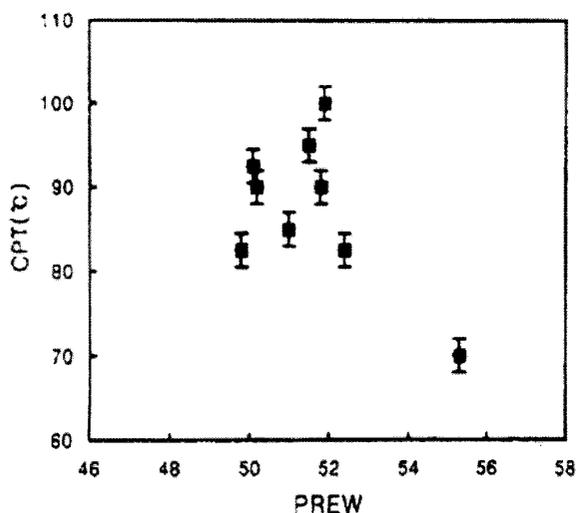


Fig. 2. Comparison between CPT's of the experimental alloy as determined by 6% $\text{FeCl}_3$  test and PREW of bulk composition.

increasing the contents of chromium and molybdenum. As if the contents of nitrogen is increased, that strengthens a passivation film. That is known and new empirical formulas have made. Rockel<sup>11)</sup> and so on said that the pitting critical temperature increased in fan shape, but Ujiro<sup>12)</sup> said that the crevice corrosion rate in  $\text{FeCl}_3$  solution is in proportion to the formula( $\% \text{Cr} + 3\% \text{Mo} + 70\% \text{N}$ ). Therefore, the opinion about how strong the effect of nitrogen has does not agree. Also, there is research result that the nitrogen has a effect with molybdenum.<sup>13),14)</sup> So the subject using PRE limits Fe-Cr-Ni-Mo-N system.

At this study, duplex stainless steel which is designed by RIST was calculated and compared the corrosion resistance of the tungsten element's alloy by Nicholles' formula( $\% \text{Cr} + 3.3\% \text{Mo} + 16\% \text{N}$ ). And at applying the material, the necessary blazing and the necessary corrosion data for the damage diagnosis are very sensitive for the environment, for the alloy structure, and for the heat-treatment. So the necessary data for the life prediction of the material must achievement the necessary corrosion test if there is a few variety of the alloy.<sup>15)-19)</sup> The corrosion progress is made progress by the function depending on the time,<sup>20)-25)</sup> and the finishing the experiment at a short of time is difficult and we estimate the rate using at least two methods from in those such as the very close experimental method setting up at the actual spot, the planning test, the electrochemical acceleration experimental method, and so on. From the test which it sees, we did the cyclic polarization test of the electrochemical acceleration experimental method, the critical pitting temperature test, and did simultaneously the local corrosion test by the immersion at the chloride environment which is very severe,<sup>26)-29)</sup> and estimated the corrosion rate and the corrosion resistance. Also we studied the effect of the corrosion resistance according to the content change of carbon, chromium and nitrogen in duplex stainless steel through the test result.

## 2. Experimental

### 2.1 Specimen preparation

For daily use of duplex stainless steel used at this experiment, the specimen that RIST designed is maintained the contents of nickel, molybdenum, tungsten, copper, manganese, and silicon, and is changed the contents of carbon, chromium, and nickel. We prepared the as-casting specimen and the water-quenching specimen after heat-treatment at 1150°C for 1hr. Preparation alloys were cut the each region after machine working and cleaning. A part cut was cleaned and the mechanical wor-

king is done by cutting a fitting size for the experiments used the electric discharge working the equipments.

The last preparation specimen is cut 2 cm\*2 cm of about 0.1cm thickness. After it cut, for removing the oil and the dirty at the surface, the specimen was cleaned for 30 minutes by the ultrasonic cleaner and treated the methyl-alcohol lastly.

After polishing at using from 20 grit paper to 2000 grit paper, and the specimen was polished by 0.3  $\mu$ m alumina powder and was cleaned. For getting the specimen of the same condition, and we did the same surface treatment. We observed the surface corrosive part in each preparation specimen at the variety methods, and we tried the eye observation, the optical microscope observation, and the secondary electrical microscope observation.

The typical parts and the strange surface were reported and the secondary electrical microscope. We observe the part which is not corroded and the cross section for each specimen and we compared and analyzed the structure pictures. The electrolysis solution is used a reagent of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , and made the 10%  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  solution more than 1 lrs by stirring, and used at testing ASTM G46 test solution. The cyclic potentiodynamic polarization test solution of the electrochemical test was made 3.4% NaCl at ASTM G61 and at this case, a NaCl reagent was used and made a equal ASTM G46 method. For testing the critical pitting temperature lastly, the 0.5M NaCl solution was made, and the reason that using NaCl of a low concentration is shown discrimination of CPT temperature of a designed specimen. We analyzed a inner pitted part and a outer element of the specimen which was observed by the optical microscope quantitatively, at this case, after observing at SEM, this part was analysis of the chloride characters.

## 2.2 ASTM G46A testing

The immersion test is the pitting corrosion resistance of stainless steel by ferric chloride solution and the specimen was putted in 10%  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  solution for 24 ~ 72 hrs (in air atmosphere, 20 ~ 50°C). After testing the experiment method for a loss in quantity and a localized attack of the specimen is the ferric chloride test that is generally the test estimating the alloy resistance of the local corrosion's spread. The immersion test is the method estimating corrosion rate by measuring a loss in weight before and after experiment and a summary graph of the test is shown Fig. 3(a). The electrolysis solution is the 10%  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  solution, and the specimen is duplex stainless steel, and the test container is a 1000ml beaker. We estimated a loss in weight of the as-cast specimen and the heat-treatment specimen finishing preparation

in the immersion test for 24, 48, and 72 hrs. So we converted that into mm/year unit.

The corrosion resistant test goals about a local corrosion is (a) the choice of alloy range and the development, (b) the destruction analysis, (c) the effect calculation according to the change of the corrosive preceding variable and (d) the prediction of corrosion rate. The specimen was estimated a loss in weight for the immersion and estimated the uniform corrosion rate, and compared a variable pitting shape (the number of pit, pitting opening size, depth of pit and so on). If the attack quantity gotten is local, the pitting problem is very serious. The accelerating test is used (a) increasing temperature and (b) increasing the concentration of the aggressive species ( $\text{Cl}^-$ ,  $\text{H}^+$ ).

## 2.3 Critical pitting temperature test

CPT is the electrochemical test of the method for the pitting experiment. At room temperature first, that is offered the anodic potential and observed a unexpected increasing current while increasing temperature, and estimated that temperature. This concept was introduced by Brigham and now used to estimate the alloy corrosion resistance for the material design and the optimization choice generally. The test method is following, the regular voltage a passivation region in anodic electric potential is impressed at a regular cell temperature, and the temperature is changed in cell until an expected current increasing at the current of a passivation region, and that temperature is used to estimate the corrosion resistance. At our test method anodic potential is fixed 200 mV, SCE in the specimen, and we test the polarization experiment in 0.5 M NaCl electrolysis solution. The reason for the choice of a low concentration electrolysis solution is expected a low CPT at a irregular specimen and that is given a discrimination at the judging of the corrosion resistant sort.

CPT temperature is increased at 5°C gap in a constant temperature container at a passivation region. We tested for about 10 minutes and considered the incubation time sufficiently before creating the pitting. These anodic polarization is continued increasing 5°C if the pitting doesn't create, and critical pitting temperature is estimated. Before a polarization the electrolysis solution is purged oxygen is the solution with nitrogen gas for 1 hr, and while test nitrogen gas is bubbled and the time increasing 5°C is taken from 15 minutes to 20 minutes.

## 2.4 ASTM G61(Electrochemical tests for cyclic polarization)

The specimen and the electrolysis solution for the electrochemical test are prepared like the preparation for

the immersion test, and the process prepared three electrode system with that specimen is following. Working electrode is made by the spot welding between 1mm diameter copper lead and specimen for polarizing the designed alloy variably. Because this method is the welding which use the resistance of the surface between two metals, the welding part is small, and the welding is done easily, and the test error for the heat is decreased. After welding the exposed surface is painted laquer and it is dry at room temperature for 3~4 hrs, and the test errors caused the surface exposure minimized.

Reference electrode is composed of 0.4 cm diameter salt bridge tip and SCE electrode in the saturate KCl solution. Counter electrode is composed of the spot-welding 99.99% Pt-falg and 0.5mm diameter of 99.99% Pt lead. The schematic graph is shown Fig. 3(b). After connecting this three electrode with potentiostat(using EG&G potentiostat/galvanostat Model 273A, M352 corrosion software), the basic mechanism study is proceeded in using the cyclic potentiodynamic method of the electrochemical experiment methods.

The method estimating the first creating a local corrosion(a pit creation) and the critical potential of the spread(the groeth of the pit) is the method searching a pitting electrochemical potential and is gotten the basic data about the local corrosion sensitivity. The test scan from  $E_{corr}$  to the initial of the local corrosion in the anodic direction and rescan reversely. At this time the current is decreased the pitting potential the protection potential and so on. Potentiostatic measurements test method impressed the electric potential in the anodic region a long time, and is useful to calculate the resistance about the start of a pit, and Galvanostatic measurement is measured the current increasing the polarization in the controlled current test about the pitting. The electric potential is also increased in proportion to the current increase such as ASTM G100.

For G-61 test the electrolysis solution is used 3.4% NaCl and potentiostat is done the cyclic polarization measurements of the software using EG&G potentiostat/galvanostat Model 273A. The test is used about 1L cell, SCE(the reference electrode) and the Pt electrode(the counter electrode) in three electrode system. Before testing, it used nitrogen gas bubbling and for the oxygen in the solution and for the estimating the corrosion resistance enable to apply at a job-site the cathodic protection was not done before testing. Scan rate is 0.17 mV/sec sweep and the initial start electric potential is switching about 1150 mV in the corrosion potential and SCE is switching the higher electric potential than the electric potential for creating the oxygen gas, and it did reverse sweep and it

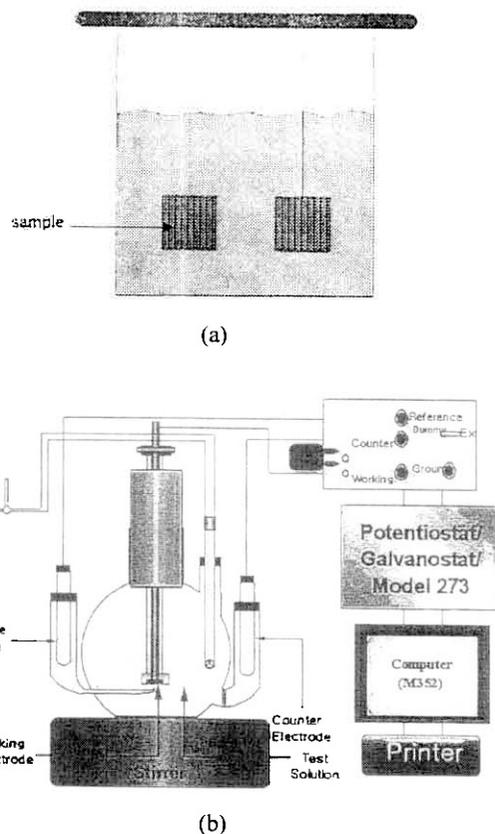


Fig. 3. Schematic experimental set-up for immersion test(a) and electrochemical test(b)

did the polarization test until the corrosion potential and it estimate the pitting electric potential.

### 3. Results and discussion

#### 3.1 The immersion test

For estimating the corrosion resistance of the specimen, it tested by immersion in 10%  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  solution, and it exposed this electrolysis solution at the air atmosphere for 24, 48, and 72 hrs, and it immersed at room temperature for comparing with several corrosion shape and rate. After the immersion test, it putted out the specimen and it removed the corrosion product on the specimen surface through flowing the clean water. Next it cleaned the specimen for 10 minutes with the ultrasonic cleaner in the acetone and after being dry for accuracy about a loss in weight it measured the weight until 0.0001g unit. It's a loss in weight is calculated mpy(mils penetration per year) or mm/year and as-cast specimen is shown Table 1 and the heat-treatment specimen is shown Table 2. The method described the corrosion rate is several but the general used method is the penetration rate and shows

**Table 1. Corrosion rate of various samples in the ferric chloride solution during 72 hours(as-cast duplex stainless steel)**

Specimen	Weight loss 72hr(g)	Corrosion rate 72hr(mm/year)
JS1	0.0027	0.336
JS2	0.0038	0.473
JS3	0.1288	14.728
JS4	0.283	35.483
JS5	0.0364	4.162
JS6	0.0026	0.296
JS7	0.0009	0.111
JS8	0.0016	0.183
JS9	0.0043	0.492
JS10	0.0053	0.665
JS11	0.0022	0.252
JS12	0.0006	0.075

**Table 2. Corrosion rate of various samples in the ferric chloride solution during different hours(heat-treatment duplex stainless steel)**

specimen	Weight difference (mg)			Corrosion rate (mm/year)		
	24hr	48hr	72hr	24hr	48hr	72hr
JS1	2.3	11.0	9.8	0.083	0.199	0.118
JS2	5.5	20.9	14.7	0.198	0.377	0.177
JS3	4.5	4.9	6.8	0.162	0.089	0.082
JS4	1.4	2.8	3.0	0.051	0.051	0.036
JS5	-0.7	1.5	-1.2	-	0.027	-
JS6	0.5	0.1	0.6	0.018	0.002	0.007
JS7	1.6	2.2	7.4	0.058	0.040	0.090
JS8	-23.28	4.0	5.8	-	0.007	0.070
JS9	9.4	17.2	28.4	0.344	0.315	0.346
JS10	4.1	5.8	9.7	0.150	0.106	0.118
JS11	1.6	2.9	8.1	0.059	0.053	0.099
JS12	0.9	1.1	1.4	0.033	0.020	0.017

mpy(mlis/year). Penetration rate is calculated easily using the following formula which the data gotten the weight method puts in.

$$mpy = \frac{534W}{DAT} \tag{1}$$

W : weight loss, mg  
D : density of sample, g/cm<sup>3</sup>

A : sample area, inch<sup>2</sup>  
T : exposure time, hours

Also, it convert the metric unit and penetration rate is following,

$$\mu m / year = \frac{87600W}{DAT} \tag{2}$$

W : weight loss, mg  
D : density of sample, g/cm<sup>3</sup>  
A : sample area, cm<sup>2</sup>  
T : exposure time, hours

Here, it is 1 mpy = 0.0254 mm/year = 25.4 μm/year. The corrosion by a loss in weight can use the basic estimate preceding data of the corrosion rate proceeded the same time the uniform and the local corrosion shape. Specially, it used a loss in weight at the immersion test and it converted equation (1) and equation (2) and it showed mm/year such as Table 2. At this case, as it is following it shows the corrosion resistance sort of the specimen in the weight decrease ratio standard.

- (1) As-cast specimen  
JS12 > JS7 > JS8 > JS11 > JS6 > JS1 > JS2 > JS9 > JS10 > JS5 > JS3 > JS4
- (2) Heat-treatment specimen  
JS6 > JS12 > JS5 > JS4 > JS8 > JS3 > JS7 > JS11 > JS10 > JS2 > JS9

Of course at this case that of the corrosion measurement methods shows the sort accurately, and the table just shows the sort change of the corrosion rate in the time function.

First it compared with the corrosion resistance by the time function in the given specimen and it knows that the sort of the corrosion resistance does not change almost in the high level but in low level the change gap of the sort changes according to the long and short of the immersion time. This reason is searched the complication which the corrosion shape is the pitting in the specimen plate and it considers the process of the pit's initiation, growth, and repassivation. We know that the defect or the impurity of the alloy but at the case of the corrosion resistant material. The initiation of the pit comes out statistically and that is already explained the variable case and conditions through the preceding study. The high level specimen is changed a little corrosion rate between the as-cast specimen and the heat-treatment specimen. After the heat-treatment the corrosion resistance of these specimen is developed, because it becomes the minuteness and the homogeneity of the structure. The more accurate explain of the mechanism developed the corrosion resis-

tance can do through the electrochemical test and the analysis of the surface. Also in the case of the immersion test as it shows the corrosion resistance change about the composition change of duplex stainless steel if PRE value is high, we get the result the corrosion resistance is good practically, at the as-cast specimen the recording corrosion resistance where the content of the carbon will be little (JS1~JS4) is bad, but after heat-treatment the corrosion resistance becomes good. The recording corrosion resistance where the content of the chromium will be much is good before and after heat-treatment all. We identify that the chromium is not influenced by the heat-treatment almost, also at the case which it added nitrogen element, the recording corrosion resistance where the content of the nitrogen will be much is good before and after heat-treatment. We identified that the nitrogen increased the corrosion resistance at all the case. As the nitrogen is the element stabilized the austenitic phase and generally we turn out the element developed the corrosion resistance, so that is the same thing that turns out through the preceding study.

At the case of the tungsten element before heat-treatment the corrosion resistance is good in proportion to the content of the tungsten but after heat-treatment the corrosion resistance is not shown certainly in proportion to the content of the tungsten and we consider that the corrosion resistance is good according to the some proper content. After the immersion test, it measured the solution and the pH change. Because the corrosion solution is not changed a lot.

This can be judged that the pollution by the corrosion of the alloy is small, so it expected that the result for this test didn't come out the experimental error. The specimen that is not shown a pit on the surface is appeared the corrosion of the degree of the little etching mainly according to the crystal grain boundary, and it is equal that the as-cast specimen doesn't treat the homogeneity.

### 3.2 ASTM G61

Table 3 is shown the electrochemical experimental value using the cyclic potentiodynamic polarization curve in the as-cast specimen. This uses the same specimen preparation and electrochemical method and it gets the result. As the data graph is duplicated to compare with and to analyze the result, it is enable to compare with the electrochemical behavior easily. Sweep rate which is the important test factor is 0.17 mV/sec, and switching potential sweeps 1150 mV, SCE that gets out of the oxygen evolution reaction to identify accurately the exist of the pitting potential the current suddenly.

Finally it reports the corrosion potential and this is the

**Table 3. Cyclic potentiodynamic polarization results for various composition**

시편	Pitting potential	Protection potential	Corrosion potential	Remark
JS1	-20mV, SCE	-120mV, SCE	-173mV	
JS2	1500mV, SCE	-280mV, SCE	-145mV	
JS3	840mV, SCE no pitting	780mV, SCE no pitting	-320mV	
JS4	350mV, SCE	-200mV, SCE	-288mV	
JS5	250mV, SCE	-80mV, SCE	-149mV	
JS7	850mV, SCE	820mV, SCE	-182mV	
JS8	170mV, SCE	-120mV, SCE	-183mV	
JS9	100mV, SCE	-180mV, SCE	-191mV	
JS10	320mV, SCE	-210mV, SCE	-273mV	

Corrosion Resistance Rank (Pitting potential의 전위 차이) : JS7 > JS3 > JS4 > JS10 > JS5 > JS8 > JS2 > JS9 > JS1

**Table 4. Experimental results for the critical pitting temperature**

시편	Applied anodic potential	Incubation time/sec	Critical pitting temperature	Current	Remark
JS1	200mV, SCE	20	55	600 $\mu$ A	
JS2	200mV, SCE	20	70	100 $\mu$ A	
JS3	200mV, SCE	150	45	20 $\mu$ A	
JS4	200mV, SCE	100	50	220 $\mu$ A	
JS5	200mV, SCE	120	50	240 $\mu$ A	
JS6	200mV, SCE	-	-	-	
JS7	200mV, SCE	120	40	120 $\mu$ A	
JS8	200mV, SCE	350	50	55 $\mu$ A	
JS9	200mV, SCE	50	25	1.2 mA	
JS10	200mV, SCE	50	25	1.5 mA	

Corrosion Resistance Rank( temperature for critical pitting ) : JS2 > JS1 > JS4 > JS5 > JS8 > JS3 > JS7 > JS6 > JS9 > JS10

initial potential of the cyclic potentiodynamic polarization and the concept sort of the corrosive thermodynamic noble and poor. But at the case of the stainless steel alloy the low and the high of the potential has nothing to do with the corrosion rate almost. It is difficult to compare with the corrosion resistant sort and it removes. The corrosion resistance sort of by the result of the protection potential and the corrosion potential just refers to decide the sort.

### 3.3 Critical pitting temperature

To decide the CPT(critical pitting temperature) it impresses the electric potential at the passivation region in

the constant temperature container for 10 minutes and the data gotten to measure the current change. Of course it is criticized a lot that the pitting initiation by increasing temperature is estimated the corrosion resistance of the designed alloy.

The typical corrosion test can be separated the acceleration test and the severe environment test. In this case it is the result that is estimated the edge of the corrosion resistance using the variable of the temperature increase which is the severe environment. So as it estimates the result, that is important test for applying at the high temperature, but it may be a restricted method as it chooses the material at the air temperature.

#### 4. Conclusions

Recently the alloy development of duplex stainless steel has been done. On this study we studied the effect of the corrosion resistance according to the composition variety of C, Cr, N in the alloy elements of duplex stainless steel.

The high level specimen is changed a little corrosion rate between the as-cast specimen and the heat-treatment specimen. After the heat-treatment the corrosion resistance of these specimen is developed, because it becomes the minuteness and the homogeneity of the structure. The more accurate explain of the mechanism developed the corrosion resistance can do through the electrochemical test and the analysis of the surface. Also in the case of the immersion test as it shows the corrosion resistance change about the composition change of duplex stainless steel if PRE value is high, we get the result the corrosion resistance is good practically, at the as-cast specimen the recording corrosion resistance where the content of the carbon will be little(JS1~JS4) is bad, but after heat-treatment the corrosion resistance becomes good. The recording corrosion resistance where the content of the chromium will be much(JS5~JS6) is good before and after heat-treatment all. We identify that the chromium is not influenced by the heat-treatment almost, also at the case which it added nitrogen element, the recording corrosion resistance where the content of the nitrogen will be much(JS9~JS12) is good before and after heat-treatment. We identified that the nitrogen increased the corrosion resistance at all the case. As the nitrogen is the element stabilized the austenitic phase and generally we turn out the element developed the corrosion resistance, so that is the same thing that turns out through the preceding study.

At the case of the tungsten element before heat-treatment the corrosion resistance is good in proportion to the content of the tungsten but after heat-treatment the corrosion resistance is not shown certainly in proportion

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