

## Electrochemical Corrosion Characteristics of Maraging Steel Welds Depending on Chromium Content

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The corrosion behavior of maraging steel weldments which depend on Cr content has been studied, and electrochemical experiments were conducted changing the immersion time in chloride solution. The study was carried out using open-circuit potential, potentiodynamic polarization, electrochemical impedance spectroscopy techniques. The results showed that corrosion rate decreased and  $E_{\text{corr}}$  and  $E_{\text{pit}}$  increased with increasing Cr content and also with increased with immersion time of the maraging steel before measurements. The surface morphology of the corroded samples has been examined by Scanning Electron Microscopy. It showed that the resistance to general and pitting corrosion increased as the Cr content increased.

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**Keywords:** Steel; polarization; EIS; SEM; Pitting corrosion

### 1. INTRODUCTION

As maraging steel is comprised of low carbon ( $C \leq 0.03$ ) Fe-Ni martensite matrix of BCC structure containing precipitation hardening alloy elements in a solid state, it is hardened by precipitation of intermetallic compounds resulting from aging at 480°C.[1] This steel of light gray color has high toughness in spite of its high strength. Many applications of maraging steels in aerospace, marine, and nuclear power fields as well as for special parts of industrial machines have been realized due to their unique combination of superior mechanical properties, fabrication, weldability and corrosion resistance.

Maraging steel was developed and industrialized by INCO at the end of the 1950s. Though development of the 20 and 25wt% Ni maraging steels was conducted at the beginning, Fe-18Ni-Co-Mo maraging steel was developed later to solve the problem of brittleness by lowering the Ni content

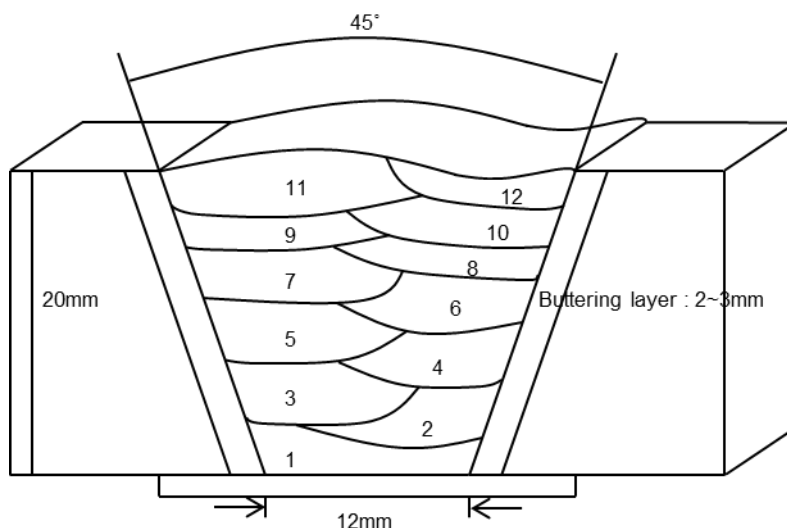
to 18% and instead adding alloy Co and Mo. However, due to rapid increase in the Co price caused by fulfillment of market demand and weighted distribution of Co resource, Fe-Ni-Mn maraging steel was developed by replacing a considerable amount of alloy Co and Ni with Mn. According to the study conducted by Hossein Nedjad, brittleness was raised as a problem because discontinuous coarse  $\Theta$ -NiMn precipitates were formed along the grain boundary of Fe-Ni-Mn maraging steel, and, to supplement this, studies on maraging steel improved with alloy elements such as Mo, Ti, Cr, W, and were actively conducted. [2-7]

Maraging steel is used in contact with acid picking, acid descaling, acid cleaning, acidizing, and so on, and, for this reason, studies on corrosion properties of maraging steel have been actively conducted. According to the literature, atmospheric exposure of 18 Ni maraging steel leads to corrosion in a uniform manner and it becomes completely rust covered. Bellanger et al.[8] reported that the effect of slightly acid pH with or without chloride ion in radioactive water on the corrosion of maraging steel, and reported that corrosion behavior of maraging steel at the corrosion potential depends on pH and intermediates remaining on maraging steel surface in the active region favoring the passivity. The effect of carbonate ions in slightly alkaline medium on the corrosion of maraging steel was investigated by Bellanger, which reveals that corrosion potential does not change with carbonate ions at a constant pH.[9] Also, it has been also reported that, as maraging steel has low diffusion of hydrogen, it is less sensitive to hydrogen brittleness than high strength steel.[10] While studies on corrosion characteristics of maraging steel in an acidic atmosphere such as sulfuric acid, phosphoric acid, and hydrochloric acid have been actively conducted recently, studies on the welded maraging steel are still insufficient.[11-16] Accordingly, in this study, the corrosion characteristics of welded maraging steel in 0.1M NaCl solution which depend on Cr addition were evaluated by conducting electrochemical experiments of OCP, potentiodynamic polarization, and EIS.

## 2. EXPERIMENTAL PROCEDURE

### 2.1. Welding Material and Method

In this study, buttering-treated maraging steel was used. The welding was conducted by FCAW method with the specimen prepared by the dimension of 300mm x 150mm x 20mm for the base metal of 304-stainless steel, and welding was carried out in 12 passes as shown in Fig.1. The root gap was 12 mm and the groove angle was 45°. Ar(75%)+CO<sub>2</sub>(25%) was used as the shielding gas considering arc stability, and the flow rate was 20ℓ/min. More detailed welding conditions are shown in Table 1. The component of filler wire was fixed with Mo, Ni and Mn and the wire was prepared by changing the content of Cr as 1.4, 5.2, 6.0, 9.3wt%. The chemical composition of welded maraging steel was measured by an Optical Emission Spectrometer (Metal-LAB75/80J, GNR srl, Italy), and the result is shown in Table 2.



**Figure 1.** Schematic Diagrams of the weldment

**Table 1.** Welding parameters

Voltage(V)	Current(A)	Polarity	Travel Speed(cpm)	Heat	Interpass
30	310	DCRP	37.7	15.4	Max. 150

**Table 2.** Chemical composition of weld metal (wt%)

	C	Si	Mn	P	S	Ni	Cr	Mo	Ti	Al	Co
No.1	0.05	0.77	0.72	0.023	0.017	12.36	1.42	3.01	0.12	0.06	0.03
No.2	0.04	0.72	0.71	0.021	0.015	11.94	5.19	3.01	0.13	0.08	0.04
No.3	0.03	0.51	0.56	0.016	0.015	12.33	6.06	3.08	0.13	0.01	0.03
No.4	0.06	0.39	0.73	0.016	0.015	11.98	9.33	2.83	0.12	0.03	0.03

2.2. Electrochemical Measurements

Electrochemical experiments were performed by using a Potentiostat Versa STAT4 (AMETEK) after varied immersion periods of 0 and 24h of welded maraging steel in NaCl solution. Specimens were ground using SiC polishing paper from #400 to #1000, and used as the working electrode (1 cm<sup>2</sup>) after going through the processes of rinsing and drying. A platinum foil was used as the counter electrode, silver/silver chloride (3mol l<sup>-1</sup> KCl, 0.197V) was used as the reference electrode, and a K0235 Flat Cell was used as the electrochemical experiment cell. The potentiodynamic current-potential curves were recorded by polarizing the specimen to -0.5V cathodically and +2.2V anodically with respect to the open circuit potential(OCP) at a scan rate of 2mV/s. The corrosion behavior of welded maraging steel was also investigated by EIS technique. The impedance measurements were carried out in the frequency range of 100kHz to 0.01Hz, at the OCP, by applying 10mV sine wave AC

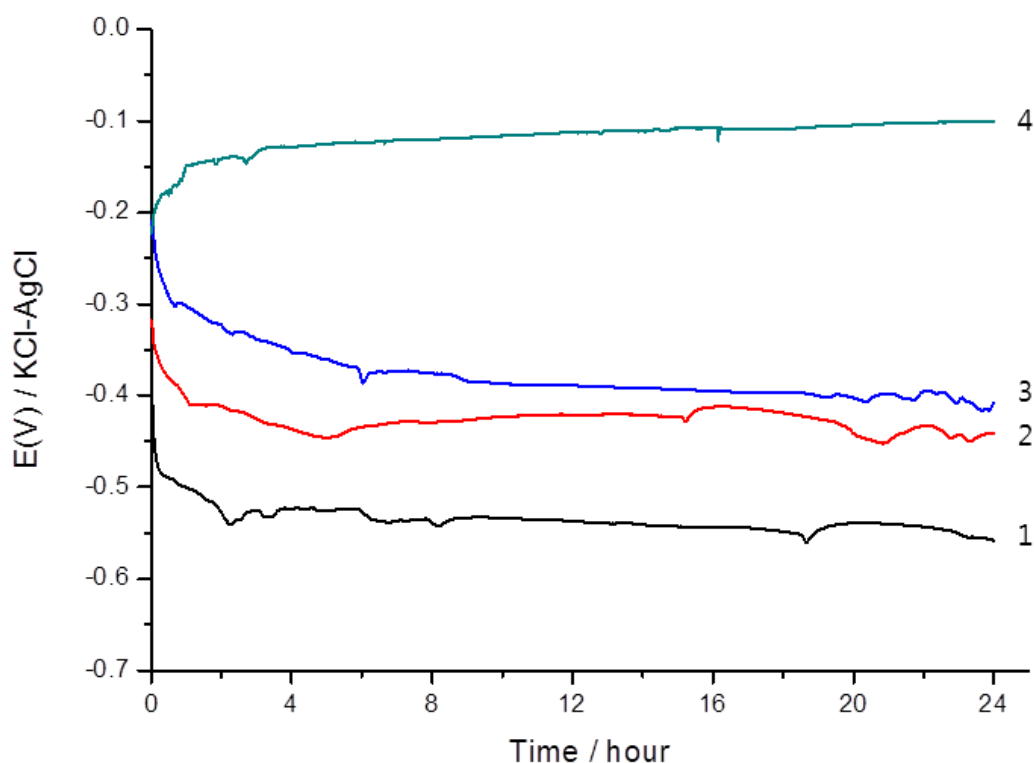
voltage. The double-layer capacitance ( $C_{dl}$ ), the charge-transfer resistance ( $R_{ct}$ ), solution resistance ( $R_s$ ) were calculated from the Nyquist plot. In all the above measurements, at least three similar results were considered, and their average values are reported.

### 2.3. Scanning Electron Microscopy (SEM)

The surface morphology of welded maraging steel after polarizing the corrosion potential ( $E_{corr}$ ) to +1.2V in 0.1M NaCl solution were compared by recording the SEM images of the samples using a Scanning Electron Microscope (JSM-6700F, JEOL).

## 3. RESULTS AND DISCUSSION

### 3.1. Open-Circuit Potential (OCP) Measurements



**Figure 2.** OCP curves for as welded maraging steels in 0.1M NaCl. (1) 1.4%, (2) 5.2%, (3) 6.0%, (4) 9.3% Cr content.

An OCP experiment was conducted in order to analyze spontaneous electrochemical reaction between the reference electrode and the working electrode in the state no current or voltage is applied. Figure 2 shows the OCP curves of welded maraging steel containing 1.4, 5.2, 6.0, and 9.3wt% of Cr respectively in 0.1M NaCl solution at 25°C. In the Curve 1, the potential of welded maraging steel containing 1.4% of Cr at the beginning of immersion time rapidly decreased in the negative direction.

This may be due to the dissolution of an oxide film was formed on the surface of the maraging steel in air. Increasing the immersion time led to dynamic equilibrium showing open circuit potential. Also, voltage fluctuation resulting from formation of pitting corrosion was observed in the process.[17] The same behavior as that in Curve 1 also appeared in Curve 2 and 3 of the maraging steel containing 5.2 and 6.0% of Cr respectively. The OCP increased to +110 and 140mV respectively in the positive direction as the Cr addition increased. Contrary to this, in Curve 4 of the maraging steel containing 9.3% of Cr, the electrode potential increased in the positive direction at the beginning of immersion time and gradually achieved dynamic equilibrium as the immersion time increased and voltage fluctuation phenomenon apparently decreased in comparison to the previous OCP curves, through which it can be seen that the resistance of maraging steel to general corrosion and pitting corrosion is improved by adding 9.3% of Cr.

In general, chromium is known to improve resistance of Fe-base alloy to general corrosion and local corrosion.[18,19] Nobuyoshi Hara reported in his study that, in the passive film formed on a Fe-Cr alloy containing 20wt% of Cr or less, Cr(III) oxyhydroxide rapidly increases along with Cr addition, due to which the corrosion resistance increases.[20] Besides, it has been verified by the studies using auger electron spectroscopy, X-ray photoelectron spectroscopy, and soft X-ray spectroscopy that the Cr content in the passive film increases as the Cr content increases.[21-23]

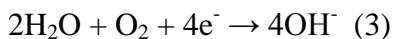
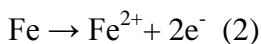
### 3.2. Potentiodynamic polarization

In order to analyze the corrosion characteristics of welded maraging steel depending on the addition of Cr, potentiodynamic polarization experiment was conducted in 0.1M NaCl solution. The potentiodynamic polarization curves of the specimens obtained by immersing the welded maraging steel in NaCl solution for 0 and 24 hours are shown in Figure 3 and Figure 4 respectively. It is clearly seen from Fig.3 and Fig.4 that increasing the Cr content in the maraging steel significantly decreased the cathodic, corrosion ( $i_{corr}$ ), anodic currents as well as shifted both the corrosion potential ( $E_{corr}$ ) and pitting potential ( $E_{pit}$ ) to more positive values. This was because the resistance to general corrosion and pitting corrosion was improved by the Cr oxide formed on the surface of the electrodes by the added Cr element.

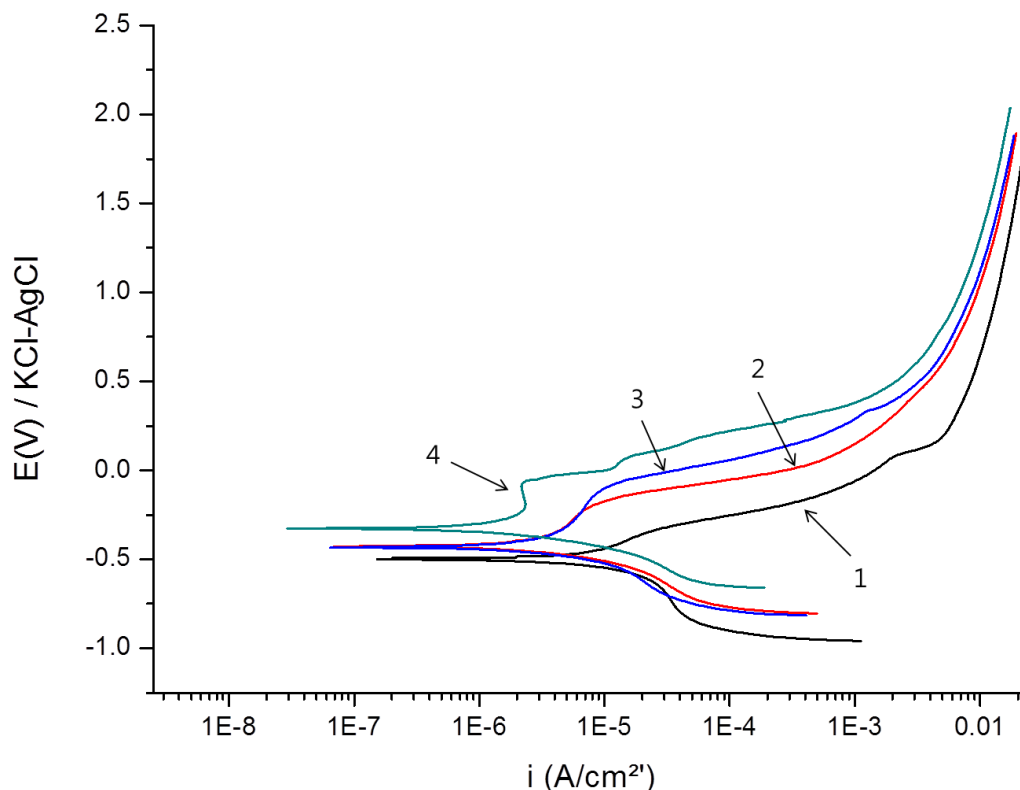
Corrosion potential, corrosion current density,  $b_a$ , and  $b_c$  which are corrosion parameters can be obtained by expanding the tafel region on the polarization curve of the two reactions, and the results of the experiment are shown in Table 3. Also, in order to calculate the corrosion rate, the measured equivalent weight ( $E_w = 2765.05, 2806.16, 2818.22, 2806.64$  grams/equivalent) and the density ( $D = 6.63, 7.65, 7.69, 7.45$  g/cm<sup>2</sup>) were entered into the software respectively, and the formula is as follows:

$$\text{Corrosion Rate(mm/year)} = \frac{0.0327 \cdot i_{corr}(\mu\text{A}/\text{cm}^2) \cdot E.W.}{\text{Density (g/cm}^3\text{)}} \quad (1)$$

When metal corrodes in an aqueous solution, oxidation and reduction reaction take place at the same time. In general electrons emitted by the oxidation reaction of iron which is an anodic reaction in metal and alloy and, simultaneously with oxidation of metal, the electrons are consumed by the reduction reaction which is a cathodic reaction.



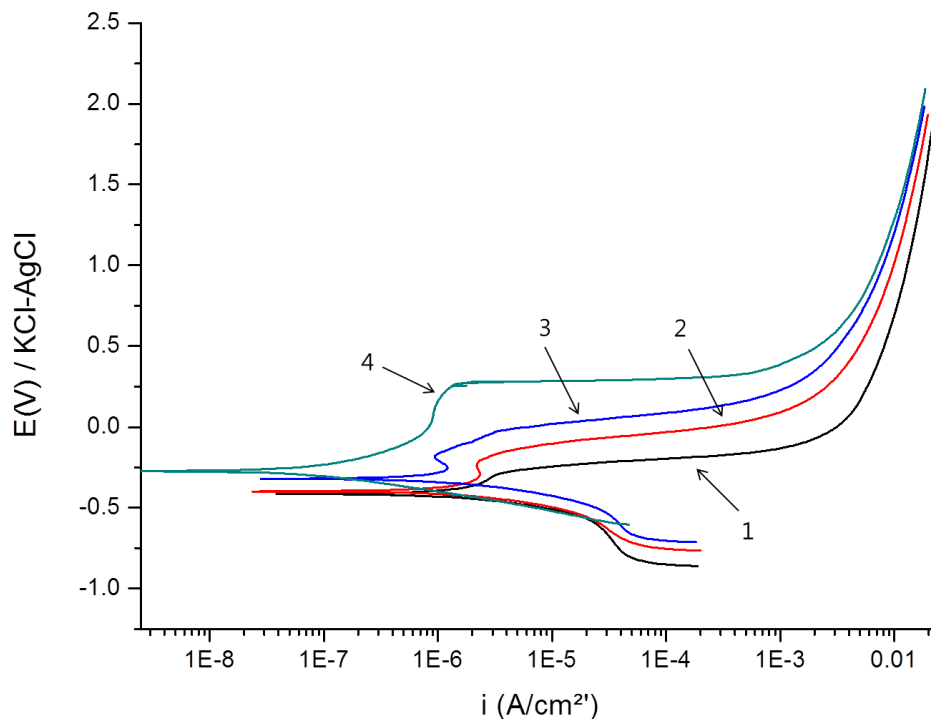
The Cr oxides resulting from the addition of Cr reduced oxidation and reduction reactions. The increase of its content reduces cathodic,  $i_{\text{corr}}$ , anodic and  $i_{\text{pass}}$  currents and corrosion rate decreased and  $E_{\text{corr}}$  and  $E_{\text{pit}}$  increased as shown in the results in Table 3.



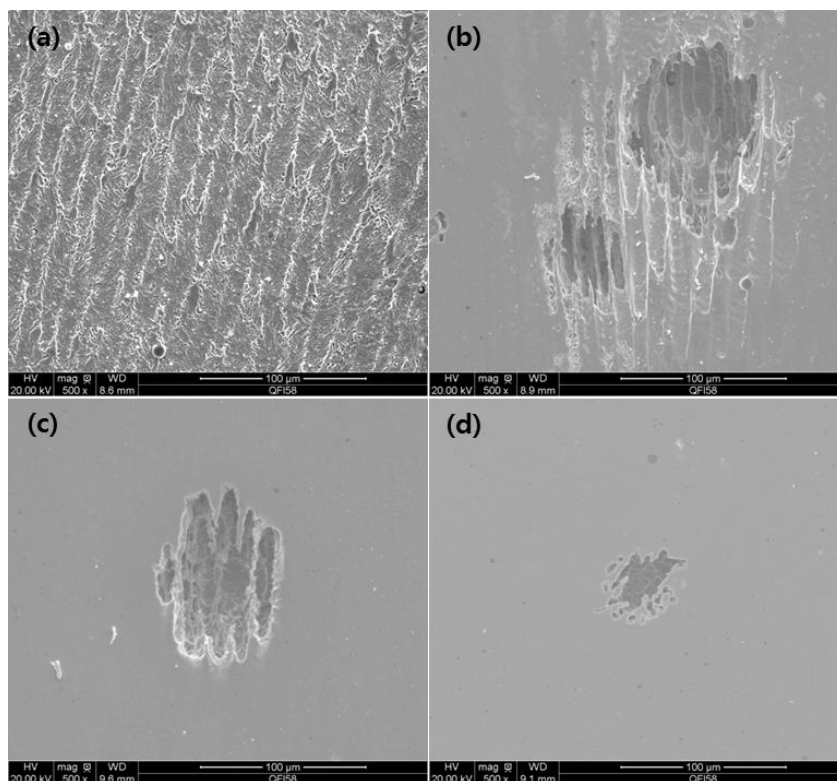
**Figure 3.** Tafel polarization curves of as welded maraging steels in 0.1M NaCl after 0h immersion in 0.1M NaCl solution. (1) 1.4%, (2) 5.2%, (3) 6.0%, (4) 9.3% Cr content.

**Table 3.** Results of tafel polarization on the as welded maraging steels in different concentration of chromium

Specimen	$E_{\text{corr}}$	$I_{\text{corr}}$	bc	ba	$E_{\text{pit}}$	$R_p$	Corrosion rate
	(mV)	( $\mu\text{A}/\text{cm}^2$ )	(mV/dec)	(mV/dec)	(mV)	( $\text{k}\Omega\text{cm}^2$ )	
No.1(0hr)	-498.94	2.018	68.47	90.26	-355.77	8.39	108.20
No.2(0hr)	-432.34	1.773	83.53	157.52	-213.16	13.39	83.69
No.3(0hr)	-428.00	1.758	87.94	157.83	-107.34	13.97	82.86
No.4(0hr)	-327.28	1.427	103.69	325.00	-92.01	23.95	69.15
No.1(24hr)	-414.20	1.285	81.83	168.96	-306.70	18.65	62.93
No.2(24hr)	-397.43	1.294	84.40	216.48	-206.10	20.40	61.08
No.3(24hr)	-320.61	1.234	90.45	343.78	-39.87	25.23	58.14
No.4(24hr)	-273.65	0.171	171.59	399.55	249.04	305.21	8.28



**Figure 4.** Tafel polarization curves of as welded maraging steels in 0.1M NaCl after 24h immersion in 0.1M NaCl solution. (1) 1.4%, (2) 5.2%, (3) 6.0%, (4) 9.3% Cr content.

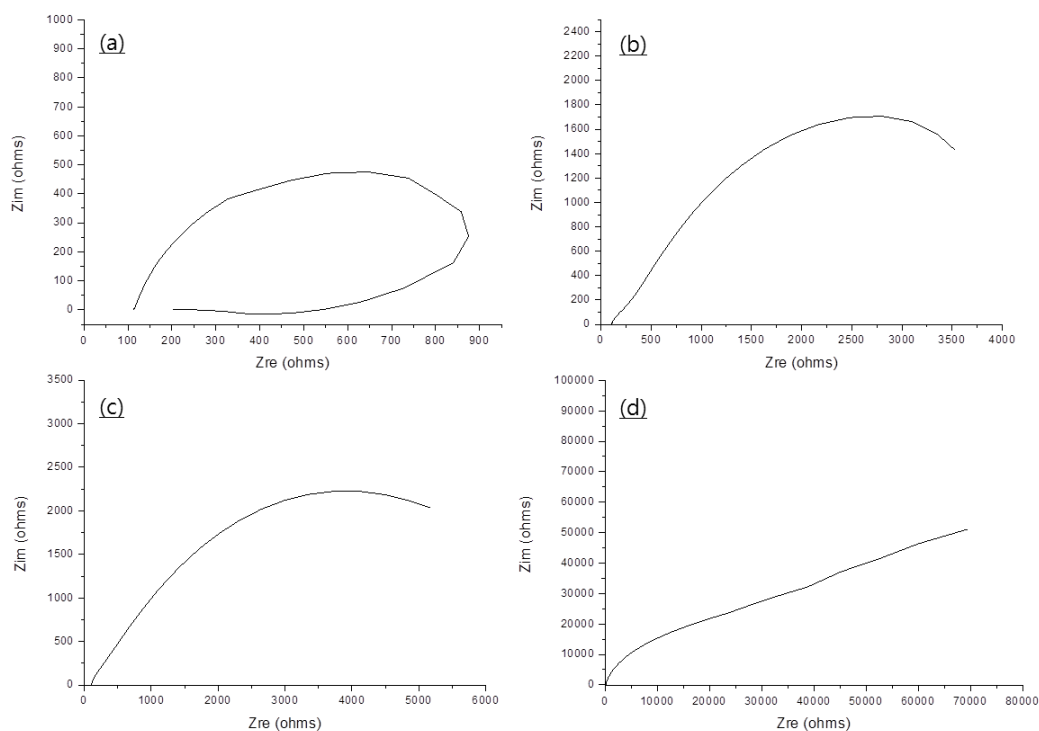


**Figure 5.** SEM images of as welded maraging steels in 0.1M NaCl (a) 1.4%, (b) 5.2%, (c) 6.0%, (d) 9.3% Cr content.

Figure 5 shows the SEM images after a polarization experiment conducted to 1.2 V (vs  $E_{CORR}$ ) in 0.1M NaCl solution. Across the whole surface of welded maraging steel containing 1.4wt% Cr, general corrosion occurred uniformly. But, on the specimens containing 5.1, 6.0, and 9.3% of Cr, no general corrosion appeared while pitting was formed, and the pitting size decreased as the Cr addition increased. Like this, it was confirmed that the resistance to general corrosion and pitting corrosion increased as the Cr addition increased, which corresponded to the results of OCP and potentiodynamic polarization experiment.

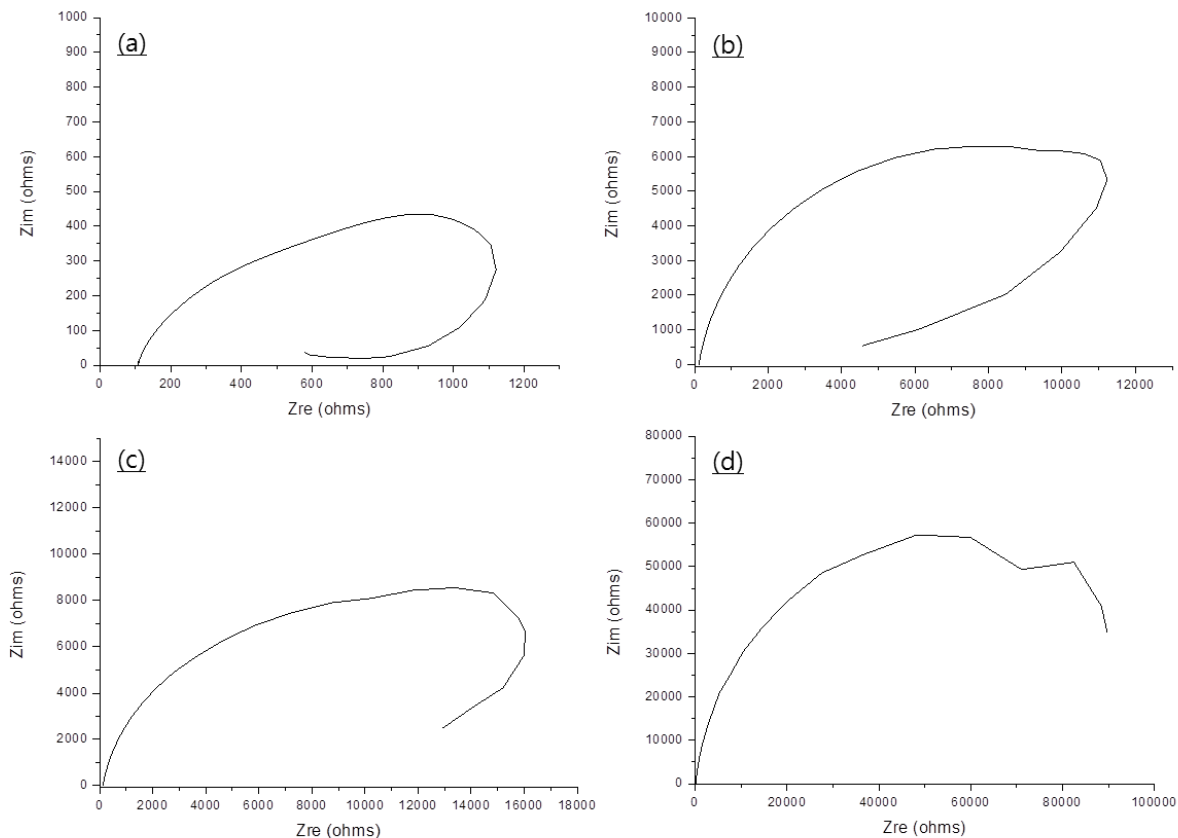
In order to determine the corrosion behavior of welded maraging steel depending on the immersion time, a potentiodynamic polarization experiment was conducted after immersing welded maraging steel in 0.1M NaCl solution for 24 hours, of which the result is shown in Figure 4. As in Table 3, Increasing the immersion time of the maraging steel alloy 1 in NaCl solution significantly decreased the cathodic,  $i_{CORR}$ , anodic and  $i_{PASS}$  currents and corrosion rate and also shifted  $E_{CORR}$  and  $E_{pit}$  to more positive values. This effect was greatly increased with the presence of Cr and up on increasing of its content in the alloy. The pitting potential of the specimen containing 9.3wt% of Cr sharply increased to +340 mV in comparison to the specimen immersed for 0 hours. This is thought to be because the resistance to general corrosion and pitting corrosion is improved by the corrosion products formed on the surface when the electrode is immersed in NaCl solution.[17]

### 3.3. Electrochemical impedance spectroscopy (EIS) measurements



**Figure 6.** Nyquist plots for the corrosion of welded maraging steel in different content of Cr after 0h immersion in 0.1M NaCl solution. (a) 1.4%, (b) 5.2%, (c) 6.0%, (d) 9.3% Cr content.





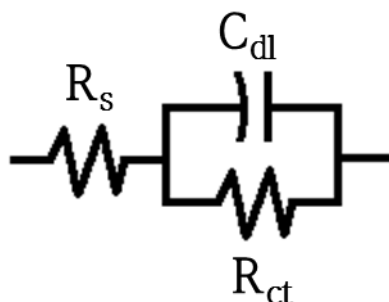
**Figure 7.** Nyquist plots for the corrosion of welded maraging steel in different content of Cr after 24h immersion in 0.1M NaCl solution. (a) 1.4%, (b) 5.2%, (c) 6.0%, (d) 9.3% Cr content.

Electrochemical impedance measurement method is known to be an effective technology for measurement of corrosion mechanism and corrosion level of metal and alloy.[24-27] In order to confirm the result of the potentiodynamic polarization experiment conducted earlier, an impedance experiment has been conducted for welded maraging steel with a different content of Cr in 0.1M NaCl solution, and the impedance results of the welded maraging steel immersed in NaCl solution for 0, 24 hours are shown in Figure 6 and Figure 7 respectively. The impedance result is shown as a Nyquist diagram. The result shows that the impedance increased as the Cr addition increased. Hamm et al [28] reported that the thickness of the passive film increases with increasing chromium content. Therefore, the corrosion rate of the alloy samples decreases. Also, in the Nyquist diagram, depression phenomena of the semi-circle were observed irrespective of the Cr addition. It can be observed that the center of the circular arc is below the x-axis like this, and it is because the Nyquist plots obtained in the real system represent a general behavior where the double layer at the metal solution interface does not behave as an ideal capacitor.[29-31]

In this experiment, an equivalent circuit model for clear analysis of impedance parameters is adopted as shown in Figure 8. The CPE is included in the equivalent circuit model considering the depression phenomenon which appears in the Nyquist diagram. The impedance parameter result fitted by the equivalent circuit model of Figure 8 is shown in Table 4. The point where the semi-circle of the

Nyquist plot intersects the real axis at high frequency yields solution resistance ( $R_s$ ). The intersection on real axis at low frequency gives the sum of solution resistance and the charge transfer resistance ( $R_{ct}$ ).  $R_s$  represents the solution resistance due to the ohmic resistances of corrosion product films and the solution enclosed between the working electrode and reference electrode.  $R_{ct}$  represents charge transfer resistance and can be defined also polarization resistance as the ( $R_p$ ) which shows the degree of electron transfer at the interface between the electrode and the electrolyte. This is inversely proportional to corrosion current density[32], and the maximum current density can be calculated through the Stern-Geary equation: [33-34]

$$i_{corr} = \frac{b_a \cdot b_c}{2.303 \cdot R_{ct} \cdot A(b_a + b_c)} \quad (4)$$



**Figure 8.** Equivalent circuits used to fit the EIS data of welded maraging steel presented in Fig.6 and Fig.7

**Table 4.** Impedance parameters for the corrosion of welded maraging steels in different content of chromium

Specimens	$R_s$ (ohm/cm <sup>2</sup> )	$C_{dl}$	n	$R_{ct}$ (ohm/cm <sup>2</sup> )
		$\mu F/cm^2$		
No.1(0hr)	70.49	106.80	0.977	941
No.2(0hr)	104.2	97.10	0.961	4,036
No.3(0hr)	108.0	85.60	0.945	6,070
No.4(0hr)	113.2	57.04	0.936	78,640
No.1(24hr)	105.1	103.50	0.975	1,132
No.2(24hr)	113.7	95.87	0.959	13,680
No.3(24hr)	114.4	80.60	0.942	16,440
No.4(24hr)	114.8	46.58	0.932	117,700

In the impedance result of test piece No. 1 containing 1.4wt% of Cr,  $R_s$  and  $R_p$  increased as the immersion time of the electrode increased. This is because the surface of the electrode was protected by the corrosion products formed as the immersion time passed increased. In the electrodes containing 5.2, 6.0, and 9.3wt% of Cr, corrosion products were also formed, and the charge transfer resistance resulting from the corrosion products was shown to be the highest in electrode No. 4 containing 9.3%

of Cr. It is confirmed that the impedance results caused by the corrosion products formed when the electrode is immersed in NaCl solution for 0 hours and 24 hours correspond to the results of the potentiodynamic polarization experiment where  $I_{\text{corr}}$  and corrosion rate decrease and  $R_p$  increases as the immersion time increases.

#### 4. CONCLUSIONS

Electrochemical experiments were conducted to evaluate the corrosion characteristics of welded maraging steel which depend on the addition of Cr and the conclusions were drawn as follows:

1) In the 0.1M NaCl solution, resistance of welded maraging steel to general corrosion and pitting corrosion increased as Cr addition increased.

2) As a result of potentiodynamic polarization experiments, cathodic,  $i_{\text{corr}}$ , anodic and  $i_{\text{pass}}$  currents and corrosion rate decreased and  $E_{\text{corr}}$  and  $E_{\text{pit}}$  increased as the Cr content increased irrespective of the immersion time. But, resistance to general corrosion and pitting corrosion improved as the immersion time increased due to formation of corrosion products.

3) In impedance experiments,  $R_{\text{ct}}$  and  $R_s$  increased and  $C_{\text{dl}}$  decreased as Cr addition and immersion time increased, which showed the same trend as the result of a potentiodynamic polarization experiment.

4) On the surface of the welded maraging steel containing 1.4wt% of Cr, general corrosion and pitting was formed. But, on the surfaces of the specimens containing 5.2wt% of Cr or more, no general corrosion was found and the pitting size decreased as the Cr addition increased.

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