# Synergistic effect of chromium content and intercritical annealing process on corrosion-resistant improvement of Ni-Cr-Mo low alloy

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## Abstract

Dual-phase steel is required for engine bolt applications due to its excellent formability without sacrificing ductility. The research aimed to investigate the synergistic effect of chromium content and the intercritical annealing process on the corrosion-resistant of low alloy Ni-Cr-Mo steel. All samples, with Cr additions ranging from 0.5-3%, were intercritical annealed at 760°C for 30 min before being quenched in water. As a result, the microstructure revealed the ferrite + lath martensite phase with a proportional volume fraction of martensite and ferrite (close 1.25 ratio). Meanwhile, the carbide particle was invisible in the SEM image. Chromium addition has a significant impact on the mechanical and corrosion properties. The martensite phase increased slightly by 2 wt.% Cr, and the hardness increased to 611 BHN, while the elongation decreased by roughly 13.8%. The highest tensile strength (~751 MPa) was obtained by adding 3 wt.% Cr. Conversely, adding chromium reduced the corrosion rate to 11.19 mmpy in the 3.5% NaCl solution.

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## **1. Introduction**

AISI 8740, also known as low Ni-Cr-Mo alloy and equivalent to 40NiCrMo2-2, is generally used for fasteners, tools joints, engine bolts, and other applications requiring high strength, fatigue resistance, toughness, and wear resistance. To achieve the optimal mechanical properties, 8740 steel for dual-phase was developed, the first generation of high-strength steel with good formability and a low YS/TS ratio. These properties are derived from a hard martensite island in the soft ferritic matrix [1]. The general composition of commercial-grade dual-phase steel, DP 500, is 0.31% Si, 0.027% Cr, 0.06% C, Fe balance [2]. The desired

microstructure and tensile strength of DP steel can be achieved using intercritical anneal [3], intercritical rolling followed by rapid cooling [4], tempering technique, or their combination [5]. Intercritical annealing is appealing work as the simplest processing for DP steels because of its continuous yielding without tensile strength losses and low costs [1]. The steel is heated above A<sub>1</sub> but below the upper critical temperature (A<sub>3</sub>) to obtain the  $\alpha$ + $\gamma$  fraction. Then, the temperature is directly reduced beneath the martensite starting temperature in order to promote martensite with diffusion-less transformation [2]. The intercritical annealing successfully produce DP structure at temperatures of 750–875°C for low carbon steel [6] and 760–810°C for low alloy steel with Mn addition [7]. Since austenite transforms to martensite, the deformed zone (plastic and elastic) generates 2–4% of volume expansion and improves the neighboring martensite [7].

The final microstructure and tensile strength of DP steels are linearly related to martensite fraction and can be influenced by element distribution. [4]. Direct data on the alloying effect in the phase transformation of DP steels are scarce. Substitution of alloying elements (Mn, Cr, and Si) or the addition of precipitate-forming elements (V, Ti, Mo, and Nb) is a promising practice for addressing the need for stronger materials. Mn, Mo, Nb, and Ta enrichment on DP steel during intercritical annealing did not completely dissolve and improved the finer dispersion of martensite, resulting in higher tensile strength, estimated 25–35 MPa per wt.% Mn [8]. The silicon concentrations ranged from 0.01–1.6%, causing cell structure to dislocate and attract growth in higher strain hardening [1]. In regards to Mn, chromium was required to stabilize retained austenite, reduce the Ms temperature, slow the kinetic of ferrite transformation, and enhance martensite formation, all of which improve the hardenability and strength [1, 6]. Filho (2016) found that the 0.6 wt.% Cr accelerated the ferrite recrystallization rate and suggested the ferrite grains refinement. Regardless of chromium content, the martensite volume increases. However, higher Cr content promoted preferentially more nucleation sites and prevented the growth of ferrite grain [9]. Martensite growing in the grain boundary of the ferrite grains. Furthermore, chromium indirectly enhances corrosion resistance.

It is important to recognize the contribution of each alloying element and heating process for the proper design of DP steels the indirectly contribute to phase transformation and mechanical properties. The primary goal of this study is to investigate the chromium effect during intercritical annealing on low Ni-Cr-Mo alloy. Intercritical annealing was performed on the sample, which was heated to 760°C for 30 min. Tensile and hardness tests are performed to describe how adding chromium affects mechanical properties. The final microstructure was established using the metallographic test.

### 2. Materials and Experiment

The steel investigated in this study was a low Ni-Cr-Mo of modified AISI 8740, which was manufactured using an induction melting furnace followed by investment casting. Table 1 shows the final chemical composition of modified AISI 8740, which was adjusted by chromium addition ranges of 1-3 wt.%. Sample A served as the main design or control

specimen. Samples B, C, and D had chromium levels of 1, 2, and 3 wt.%, respectively. The ingots were homogenized at 1100°C for 3 h to remove the dendritic structure. All samples with dimensions of  $150 \times 10 \times 10$  mm were hot forged and intercritical annealed at 760°C for 30 min in a muffle furnace, followed by fast quenching. JmatPro simulation was used to calculate the lower and upper critical temperatures (A<sub>1</sub> and A<sub>3</sub>), 734–766.4°C, respectively.

	Sample				
Composition (%)	Α	В	С	D	
С	0.44	0.44	0.41	0.42	
Si	0.40	0.37	0.35	0.29	
Mn	0.85	0.83	0.76	0.75	
Р	0.012	0.012	0.012	0.010	
Cr	0.598	1	2	3	
Ni	0.4273	0.4215	0.4422	0.4228	
Мо	0.3236	0.3234	0.308	0.3277	

 Table 1. Composition of Modified AISI 8740.



Figure 1. Schematic diagram of DP Process.

Each sample was cut to  $1 \times 1$  cm<sup>2</sup> dimension and mounted with epoxy resin. Before the metallography test, specimens were wet-ground with SiC (80–2000 grit size), polished with alumina slurry, and etched in 2% Nital solution. The microstructures were determined by using Olympus DP-12 optical microscope and JEOL JSM-6390A electron microscope. The volume fractions of martensite and ferrite were calculated according to ASTM E112-point

count method. Mechanical properties were evaluated using hardness (HM-200<sup>®</sup> Series Micro Vickers Hardness Testing Machine) and tensile tests (Tinus Olsen 300SL) as per ASTM E8M standard. The microhardness was tested at 10N load, and 10s dwell time of parameters, with 5x repetition.

The potentiodynamic polarization test was performed using the Gamry system. Three electrodes cell was composed of a specimen as a working electrode, an SCE as a reference, and a Pt wire as a counter electrode. The corrosion samples were prepared the same way the metallographic test have done. Before scanning, a 1.00 cm<sup>2</sup> surface area was immersed in a 3.5% NaCl solution at room temperature.

## 3. Result and Discussion

Figure 2 reveals the optical and SEM images of Modified AISI 8740 microstructures, which consists of dual-phase microstructures with a fraction volume balance (close to 50%:50%) after intercritical annealing at 760°C for 30 min. Samples A-D have a similar structure with uniform distribution of the ferrite phase (bright area) and irregularly shaped lath martensite (dark phase) without the Cr-rich carbide form. The ferrite microstructure gets smaller with increasing Cr content, transforming the equiaxed ferrite to a lath-like morphology. During heat treatment, the chromium and carbon were mostly dissolved into the ferrite matrix phase, suggesting the formation of uniform dislocations in recrystallized ferrite [10]. During the intercritical annealing process, ferrite + pearlite was transformed to ferrite + austenite, and further, austenite would be transformed to martensite via a diffusion-less process [11]. However, porosity was observed in all samples due to the imperfect casting process.









(d)

**Figure 2.** Microstructure of the modified AISI 8740 in OM and SEM with backscattered electrons (BSE): (a) 0.5 wt.% (b) 1 wt.% (c) 2 wt.% (d) 3 wt.% intercritical annealed at 760°C.

Furthermore, Energy Dispersive Spectroscopy (EDS) revealed that the martensite lath contained a higher chromium content than ferrite. Points 1 and 2, represented as ferrite in Figure 2a, have a 0.6 wt.% and 0.77 wt.% C, respectively. Meanwhile, the chromium content on points 3 and 4 was 0.83 wt.% and 0.68 wt.%, respectively. Carbon was found to be more abundant in martensite and M/F interfaces than in ferrite, whereas chromium was dissolved preferentially in the matrix phase and tended to become martensite [11, 12]. Carbon provided austenite stabilization and grain size improvement [7].

Parameter	Sample			
	#A	# <b>B</b>	#C	# <b>D</b>
Martensite (V <sub>m</sub> . %)	44.56	46.55	58.12	57.48
Ferrite (V <sub>f</sub> . %)	55.44	53.45	41.88	44.52

 Table 2. Volume Fraction of Microstructure in Modified AISI 8740 (Image J Simulation).

Table 2 presents the volume fraction of martensite ( $V_m$ ) and ferrite ( $V_f$ ), estimated by ImageJ software. The  $V_m$  was slightly increased to 55.48%, with a 3 wt.% increase in chromium content. At a higher temperature of 800°C, the advanced chromium content in

order more than 0.6 wt.% provided the driving force to increase the recrystallization rate while simultaneously preventing ferrite growth [9].

#### Mechanical properties of DP steel

Dual-phase steel is a high-strength low alloys (HSLA) type whose mechanical properties are attributed to the ferrite and martensite volume fraction. Figure 3 depicts the hardness and tensile properties of the sample under consideration. According to Figure 3a, increasing chromium content up to 2 wt.% was accompanied by increasing in the hardness result, ranging from 558 to 635 BHN. A tremendous increase in the DP steel hardness was caused by the severe lattice distortion and the inhibition of dislocation movement. However, in the presence of 3% Cr, the decrease in hardness was associated with a rise in yield Strength (497.7 MPa). Outwardly, there are synergistic effects of potential alloy (Cr) content within martensite which can eliminate residual stress by volume expansion (about 2–4%) from transforming martensite to ferrite, or martensite becomes soft. As a result, homogeneous plastic deformation, delayed strain localization, and reduced hardness has occurred [11, 13].



**Figure 3.** Effect of chromium content on (a) hardness and (b) tensile properties of modified AISI 8740.

Based on Figure 3b, increasing the chromium level reduced the elongation (from 29.9% to 13.8%) and slightly increased the strength (from 523 MPa to 751 MPa). The yield point of elongation in all four samples resulted in continuous yielding [13]. Sample A had the highest elongation value (29.9%) when no more chromium was added. The ductility of DP steel was related inversely to its strength. It could be attributed to an increase in the martensite fraction, and it has been confirmed that chromium encourages the faster kinetic rate of austenite formation. The greater the volume of austenite, the greater the volume of transformed martensite after rapid cooling, affecting the steel strength and elongation reduction [8]. The decreasing yield strength (YS) value denotes chromium prevents dislocation on elastic deformation. When chromium was added, steel that was produced by the intercritical annealing process became more brittle, thus indirectly affected on the

dislocation mechanism. Furthermore, the low elasticity of DP steel was caused by the absence of austenite retained (RA) in the microstructure formation. The austenite phase provides good elasticity and high toughness [9].

#### Corrosion behavior of DP steel

A potentiodynamic test was used to investigate the corrosion behavior of DP steels in 3.5% NaCl solution. The graph in Figure 4 displays the log of current density (*I*) vs. potential (*E*). The curves were plotted and fitted by linear extrapolation to obtain corrosion parameters, such as corrosion potential ( $E_{corr}$ ), corrosion current density ( $i_{corr}$ ), anodic Tafel constants ( $\beta_a$ ), and corrosion rate ( $C_R$ ), which are presented in Table 3. All samples were immersed for 1 hour before the test to stabilize the open circuit potential ( $E_{ocp}$ ) in cell corrosion. An oxygen reduction reaction ( $O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$ ) was dominated in the cathodic branch, while the anodic branch is related to the dissolution reaction of Fe [14].



Figure 4. Polarization Result of Modified AISI 8740 in 3.5% NaCl solution.

As shown in Figure 4, samples C and D had a thin passivation layer and redissolution behavior, indicating the possibility of pitting corrosion. The corrosion anodic current increased slightly in the 3% Cr sample graphs and indicated the re-dissolution when it reaches 0.4 volts. The passivation occurred at -650 mV for sample C and -620 mV for sample D, followed by re-dissolution at around -530 mV, which could be due to the breakdown of a passive layer [14]. The chloride ion penetrates the metal surface when exposed to chloride solution, causing Fe and Cr dissolution and tending to complex ferric chloride solution [15]. The curves shifted to the upper left, resulting in higher positive corrosion potential ( $E_{\text{corr}}$ ) and lower current density ( $i_{\text{corr}}$ ). As the chromium content increased, the corrosion resistance increased to ~11.19 mmpy, leading to  $E_{\text{corr}}$  values of -695.2 mV, -687.4 mV, and -682.2 mV for 1, 2, and 3 wt.% Cr, respectively (Table 3). The  $i_{\text{corr}}$  values ranging from 0.02 to 2.212  $\mu$ A/cm<sup>2</sup> were also noticed to be lower than those

obtained by Salamci *et al.* [16]. Salamci *et al.* reported that the  $i_{corr}$  value of DP steel with martensite volume fraction of 25–35% was 2.11–14.5  $\mu$ A/cm<sup>2</sup>. The ferrite phase acts as an anode, and the martensite as a cathode leading to micro-galvanic corrosion in the constituent phase of DP steel [11, 16]. The martensite/ferrite ratio change would be related to the anode/cathode area ratio.

Consequently, increasing the M/F ratio reduced the corrosion rate of DP steel [11]. Since the intercritical annealing process produces a more refined structure (Figure 1), the current value will decrease due to the more extensive ferrite/martensite interface. Fushimi *et al.* [17] also observed a lower corrosion rate due to the higher carbon solubility in austenite and reduced residual stress from martensite transformation, both of which reduce the overpotential for hydrogen reaction. Martensite was more resistant to chloride solution due to the higher carbon levels. Ferritic-martensitic steel is more resistant to corrosion than ferritic-pearlitic steel.

Parameter	Sample A	Sample B	Sample C	Sample D
Beta (mV/decade)	123.5	93.55	65.36	63.21
$E_{\rm corr}~({ m mV})$	-753.6	-695.2	-687.4	-682.2
$i_{\rm corr}~(\times 10^{-6}~{ m A/cm^2})$	2.212	0.954	0.557	0.0245
Corrosion rate (mmpy)	101.1	43.58	25.42	11.19
Passivity	No	No	Small (-650 to -600 mV)	Small (-620 to -530 mV)

**Table 3.** Effect of Chromium Content on Corrosion Behavior of DP Steel.

## EIS Study

Electrochemical impedance spectroscopy (EIS) data was obtained from a small amplitude of AC signal in a specific frequency domain that was applied to the corrosion cell. EIS represents the response of different components, such as resistance of electron charge transfer ( $R_{ct}$ ), double layer capacitance ( $C_{dl}$ ), solution resistance ( $R_{s}$ ), and Constant Phase Element (CPE, which also denote the capacitance of film and pores resistance in the corrosion film) [18]. CPE compensates for the semicircle arc of frequency dispersion related to the surface inhomogeneity and surface roughness. The EIS parameter was obtained by fitting with the Electrical Equivalent circuit (EEC) model to understand the corrosion mechanism in the metal interface. The EIS spectra and fitting results were displayed in Figure 5 and Table 4, respectively. Warburg impedance was seen in all samples.



Figure 5. (a) Nyquist plot, (b) electric equivalent circuit, and (c) Bode plot.

The Nyquist plot in Figure 5a shows that the Cr addition in dual-phase steel improved the angle radius of the capacitive semicircle and significantly increased the length vector  $|Z_{imag}|$  impedance value. The change of the |Z| value indicates the higher electron-charge transfer resistance ( $R_{ct}$ ), capacitive behavior, corrosion kinetic, and following the potentiodynamic result [19]. The  $R_{ct}$  was proportional to oxide film resistance and inversely proportional to the corrosion rate. With increasing the chromium content to 2 wt.%,  $R_{ct}$  was higher than the others, around ~39.7±0.25  $\Omega$ , exhibiting higher film compactness. On the contrary, a 3 wt.% Cr provided a larger oxide layer thickness resulting in a lower CPE value, 652.7±22.41 µS·s<sup>n</sup>, which reduced the corrosion resistance to ~11.19 mmpy (Table 2) [19].

Sample	$R_{ m s}\left(\Omega ight)$	Rct	CPE $\mu S \cdot s^n$	n	$W(\mathbf{mS}\cdot\mathbf{s}^{1/2})$	Goodness of Fit
А	901.5±4.7	$5.23 \pm 0.03$	1406±26.1	0.706	1042±43	$5.638 \cdot 10^{-3}$
В	954±6.4	$11.64 {\pm} 0.07$	$1105 \pm 25.8$	0.734	$5.356 {\pm} 1.03$	$5.579 \cdot 10^{-3}$
С	$1543 \pm 19.1$	39.7±0.25	$1415 \pm 44.03$	0.618	51.38±3.39	$4.651 \cdot 10^{-3}$
D	$1217 \pm 11.2$	$36.25 \pm 0.2$	652.7±22.41	0.702	$1.77{\pm}0,2$	$2.456 \cdot 10^{-3}$

Table 4. EIS parameters obtained by fitting with the electric equivalent circuit.

Bode plots in Figure 5b represent the impedance modulus and its phase-angle. In low frequency (0.1–10 Hz), the impedance phase of Sample #A decreased to  $-50^{\circ}$ , further increased close to  $0^{\circ}$  in moderate frequency (10–1000 Hz), and constant  $0^{\circ}$  in high frequency (1–100 kHz). It has indicated the capacitive behavior and leads to improving the corrosion behavior through passivation with Cr<sub>2</sub>O<sub>3</sub> protective films [11].



(a)

(b)

**Figure 6.** SEM image of corroded metal surface: (a) sample C and (b) sample D after polarization.

The rust formation on the metal surface of samples C and D after polarization was evaluated using SEM and depicted in Figure 6. There is a compact corrosion product with a non-uniform thickness (Figure 6a) and pit formation (Figure 6b). The addition of chromium hinders magnetite's nucleation and promotes goethite, which is beneficial to change the anodic current density and decreasing the corrosion rate [20]. Moreover, chloride ions might penetrate the rust and continue corrosion beneath the metal surface [21].

## 4. Conclusion

The synergism between chromium addition and intercritical heating successfully produced dual-phase (lath martensite + ferrite) microstructure in low Ni-Cr-Mo alloys. The chromium addition of up to 2 wt.% significantly affects the martensite fraction and prevents ferrite growth at high temperatures. The strength has been found to increase from 523 MPa

(0.5 wt.% Cr) to 751 MPa (3 wt.% Cr) to maintain the continuous yield close to 500 MPa, leading to the higher ratio of YS/TS and inversely reducing the elongation to 13.8%. Corrosion behavior is greatly influenced by the martensite fraction (which is controlled in micro-galvanic corrosion) and steel composition (which depends on passive film formation). With 2 wt.% Cr addition, the tendency of metastable passivation has been evidenced by slowly increasing the current density at -650 mV (Sample C), a lower  $R_s$  value (901.5±4.7  $\Omega$ ), and a higher  $R_{ct}$  value (~39.7±0.25  $\Omega$ ). The ferrite phase acts as an anode and the martensite as a cathode leading to micro-galvanic corrosion.

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