Effect of N on corrosion resistance of Fe-Cr-Ni-Mo-Mn alloy

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Abstract

In order to develop the Fe-Cr-Ni-Mo-Mn-N corrosion resistant alloys, analyze the influence of nitrogen on the corrosion resistance of Fe-Cr-Ni-Mo-Mn alloy, adjust the N content in the alloy system, and Flux cored wires with different N content were prepared. They were surfaced on low carbon steel by MIG welding. The phase composition, microstructure and corrosion resistance of the cladding metal were analyzed to study the effect of N content on the structure and performance of the surfacing metal. The results show that the addition of nitrogen does not change the matrix structure (which is Fe-Ni-Cr austenite), but with the increase of nitrogen content, the precipitation of nitrides (Cr2(C,N) and BN) is accompanied; when the amount of nitrogen added reaches 1.0%, the corrosion resistance of the surfacing metal is the best. At this time, the self-corrosion current density is at least 1.569 × 10^{-9} mA cm^{-2} and the self-corrosion potential is at most −0.318 V. The addition of nitrogen can promote the formation of austenite and nitride, inhibit the appearance of carbides, avoid the phenomenon of poor Cr, and improve the corrosion resistance of the material.

1. Introduction

With the rapid development of society, people’s demand for various materials is increasing, which makes the petrochemical industry develop rapidly. Since most of the petrochemical industry’s production process is in contact with corrosive raw materials or products, this makes the materials used in the petrochemical industry have stricter requirements for corrosion resistance. Therefore, stainless steel is the most commonly used material in the petrochemical industry, including common austenitic stainless steel, ferritic stainless steel, and duplex stainless steel. These stainless steels have different application fields due to their different characteristics. A large number of scholars have carried out researches on the corrosion resistance of stainless steel and have reached many constructive conclusions. Zou [1] studied the phase transformation and its effects on mechanical properties and pitting corrosion resistance of 2205 duplex stainless steel. The results shows that σ phase markedly increased the hardness and decreased the impact toughness of the test steel and during the incubation period of σ phase nucleation. The pitting corrosion test temperature and the aging time had collaborative effects on evidently displacing the pitting potential towards less noble values. Basak [2] studied the hardness, wear and corrosion resistance of 316L by electron beam surface treatment. The results show that the surface microhardness of the EB treated material is 22% enhanced , the corrosion potential is improved and order of magnitude reduced in equilibrium corrosion current density. Wang [3] studied the corrosion resistance of selective laser stainless steel via cryogenic treatment. The results show that the pitting potential and impedance value gradually increased after cryogenic treatment and the passivation film contained more Cr2O3 after cryogenic treatment. Yang [4] printed the austenistic stainless steel on carbon steel with additive manufactured component. With the arc current increasing from 140 to 180 A, the microstructure transform into coarse-grained austenite, low-quantity ferrite and new-generated a phase, the corrosion resistance decrease.

However, no matter what kind of stainless steel, Cr and Ni are mainly added to improve the corrosion resistance, and different structures can be obtained by adjusting the content of Cr and Ni. As the main additive element in stainless steel, Ni is widely used. However, the global nickel resources are unevenly distributed, and
the price of nickel increases year by year. Therefore, from the perspective of technological development, it is very necessary to develop low nickel stainless steel to reduce nickel digestion. Nitrogen is an austenitizing element, which has the effect of expanding and stabilizing the austenite in steel, and it is 25–30 times that of nickel. Therefore, many scholars have conducted research on high-nitrogen steel or high-nitrogen low-nickel stainless steel. Under normal pressure, the solubility of nitrogen in liquid iron is very low, which limits the production of high nitrogen steel. Therefore, the current production of high nitrogen steel mainly adopts pressure smelting method \[5, 6\], or by adding a certain amount of Cr/Mn element, in order to improve the solubility of N element \[7\]. Adding nitrogen to stainless steel can reduce the digestion of Ni, and can improve the corrosion resistance of stainless steel, especially in the three corrosion conditions of pitting, knife-line corrosion and intergranular corrosion in chloride ion environment. Zhou \[8\] studied the corrosion resistance of 316 austenitic stainless steel with increasing nitrogen and decreasing nickel in river water. The results show that the corrosion resistance of improved 316 stainless steel with increasing nitrogen and reducing nickel is better than that of traditional 316 stainless steel. The polarization curve and the fitting parameters of AC impedance spectroscopy reflect the same law. After more than 30 days, the passive film on the surface of the test steel is destroyed and the corrosion rate increases. H. Hänninen \[9\] found that nitrogen alloying had a well defined and marked improving effect on mechanical and corrosion properties of these steels until the solubility limit of N is reached and nitride precipitation exhibits its deleterious effects on these properties. It is also demonstrated that N contributes significantly to sliding wear and cavitation erosion resistance of stainless steels. With the development and application of high-nitrogen stainless steel, the welding process and materials of high-nitrogen stainless steel have also been developed rapidly. O.Kamiya \[10\] studied the microposity formation in partially melted zone during welding of high nitrogen austenitic stainless steels. LIU \[11\] studied the microstructure and mechanical properties of the high nitrogen stainless steel weld by adding nitride to the molten pool. Although there were no special welding materials for high-nitrogen stainless steel in the early stage \[12\], as the demand drew, scholars have started the development of high-nitrogen steel welding materials. LIU \[13\] studied the mechanical properties change under different content of nitrogen. The results show that highest mechanical properties has been obtained when 0.6% nitrogen. But for stainless steel, its corrosion resistance has an important impact on its usability. Therefore, this article takes Fe-Cr-Ni-Mo-Mn alloy as the research object, adjusts the N content, and studies the influence of N on the corrosion resistance of this austenitic stainless steel. At the same time, the distribution form of N element and the mechanism of its influence on corrosion resistance will be explored.

2. Experiments

The Cr, Mn, Mo, Ni used in the test are high-purity alloy powders, and their purity is greater than 99.9%. N is provided by CrN with 18% N content and 61.5%Cr. B is provided by Ferro Boron with 18% B. In order to prevent the alloy powder from absorbing moisture and causing defects such as pores and cracks in the flux-cored wire welding process, all powders must be dried before configuration. The drying temperature is 150°C, and the drying time is 1h. The content of N in flux-cored wire is 0.2, 0.4%, 0.6%, 0.8%, 1.0%. In the experiment, the deposited metal is prepared by welding, and excessive addition of N element will lead to porosity. By
continuously adding the content of N element, it is found that when the content of N element increases to 1.0%, pores appear in the deposited metal, as shown in figure 1. Therefore, the addition of N element is set to 1.0%. The specific alloy composition is shown in table 1. After these alloy powders are uniformly mixed, they are placed in the S304 steel strip to prepare a \( \Phi 2.4 \) mm flux-cored welding wire. The S304 steel strip is 16mm wide and 0.35mm thick, and its composition is shown in table 2. The filling rate of flux-cored wire is set to 40%. After the flux-cored welding wires were made, they were surfaced on low-carbon steel with MIG. The welding current is 200A, the welding voltage is 24V, the welding speed is 10 cm min\(^{-1}\), and the gas flow rate is 12 l min\(^{-1}\).

After welding, the welded plate was processed into performance samples and microstructure samples by mechanical processing. The phase composition, microstructure and corrosion resistance tests were carried out respectively. The phase composition was tested by XRD, and the testing condition parameters were: pure copper target, voltage 40 KV, current 30 mA, step length 2 degrees/min, scanning range 20° to 90°. Microstructure and element distribution were detected by GeminiSEM 300 with EDS. The electrochemical corrosion was carried out in 3.5% NaCl solution with different nitrogen additions, and the Tafel curve and open circuit of the surfacing layer samples were measured through the CS electrochemical workstation. Pitting related information such as potential and AC impedance spectroscopy. In this paper, three experiments are repeated to ensure the reproducibility of the experimental data. At the same time, in order to ensure the representativeness of the test data, three samples are used for each group of tests, and the test results are their average values.

### 3. Experimental results and discussion

In order to understand the influence of the N element content on the phase composition, the phase compositions of the surfacing layer are analyzed under different N contents, and the results are shown in figure 2. It can be seen from figure 2 that the phase compositions of the surfacing layer still have a certain changes

![XRD diffraction pattern of different N addition amount.](image)

**Figure 2.** XRD diffraction pattern of different N addition amount.

### Table 1. Content of each element in alloy powder (mass fraction, wt%).

| Sample No. | N  | Cr | Si  | Mo | Ni | B  | Mn | Fe |
|------------|----|----|-----|----|----|----|----|----|
| N-1        | 0.2| 22 | 4.5 | 2  | 10.6| 1.6| 10 | Bal|
| N-2        | 0.4| 22 | 4.5 | 2  | 9   | 1.6| 10 | Bal|
| N-3        | 0.6| 22 | 4.5 | 2  | 7.4 | 1.6| 10 | Bal|
| N-4        | 0.8| 22 | 4.5 | 2  | 5.8 | 1.6| 10 | Bal|
| N-5        | 1  | 22 | 4.5 | 2  | 4.2 | 1.6| 10 | Bal|

### Table 2. Composition of 304 steel strip (mass fraction, wt%).

| Element | C     | Si | Mn | Ni | Cr | Fe |
|---------|-------|----|----|----|----|----|
| Content | <0.08 | <1 | <2 | 8  | 18 | Bal|
under different N content. Although the matrix is austenite (Fe-Cr-Ni), the content of BN, M₂B (M is mainly Fe, Cr and Mn elements) and Cr₂(C,N) in the surfacing layer changes with the change of N content. Because the content of N element is too small, it is difficult to find the strict corresponding relationship between phase composition and N content from XRD results.

The metallographic structures of the surfacing layer with different nitrogen additions are shown in figure 3. It can be seen from figure 3, in the deposited metal without N element, in order to determine the composition of irregular precipitates produced during welding, energy spectrum analysis is carried out as shown in figure 3 (a), as shown in table 3. By analyzing the composition of point a and calculating the atomic percentage, it is found that the atomic ratio of Cr element to C element in the circular phase of point A is 23:6, so the phase of point B is
determined as $\text{Cr}_23\text{C}_6$. As a surface active element, n is preferentially segregated at the grain boundary, which inhibits the formation and precipitation of $\text{Cr}_23\text{C}_6$. Although the morphology of the grains changes under different nitrogen content, they are all composed of a gray-white matrix and dark gray dendrites. Combined with the XRD analysis results, the dark gray structure should be Fe-Cr-Ni austenite matrix, and the gray-white structure should be the precipitated hard phase, mainly including borides and nitrides. These hard phases mainly present a grid-like and fishbone-like distribution. With the gradual increase of nitrogen content, the matrix structure does not change much, but the size of the precipitates gradually increases. When the nitrogen content reaches the maximum at 1.0%N, both the matrix and the precipitated phase show a trend of refinement, the result is shown in figure 3f. As an austenite element, nitrogen element can increase the stability of undercooled austenite in the alloy to a certain extent, so that the austenite will not undergo other transformations during the cooling process. However, when the amount of nitrogen added gradually increases and exceeds its maximum solid solubility, the excess nitrogen atoms will be precipitated in the form of precipitated phases. When Cr is combined to form $\text{Cr}(\text{C}, \text{N})$, the corrosion resistance of surfacing layer can be improved.

In order to clearly reflect the respective positions and morphologies of the phases in the surfacing layer, the 1.0% N surfacing layer was tested by EDS, and the results are shown in figure 4. Tests are performed on representative areas in the microstructure, and the data are shown in table 4. Combining the material element content in table 1 and the test data in table 4, it can be found that the A/B area in figure 3 should be the matrix.
Taking into account the problem of EDS detection accuracy, there is basically no B element in the A/B test positions, and the other elements are basically close to the theoretical value except for Cr. Therefore, A/B should be the Fe-Cr-Ni matrix. The atomic percentage of boron at point E reaches 49.65%. Combined with the XRD results, it is inferred that the substance here is M2B. The element content at C/D/F is relatively close, but there are more elements, and the regularity is not very strong. Combined with the XRD data, it is speculated that the compounds at these positions are corresponding to the composite compound formed by CBN.

For austenitic stainless steel, its most concerned performance should be its corrosion resistance. The corrosion resistance directly affects the service cycle and safety of its products. The above analysis shows that the addition of nitrogen changes the phase composition, which will inevitably affect its macroscopic mechanical properties and corrosion resistance. Therefore, electrochemical corrosion is performed on the surfacing layer under different nitrogen content, and the polarization curve is shown in figure 5. It can be seen from figure 5 that the shape of the polarization curve of the surfacing layer under different nitrogen content is basically the same. Judging from the shape of the curve, it is basically an anodic control type. With the increase of N content, the corrosion potential shows an increasing trend, and the maximum value is obtained at 1.0% N (−0.318V). The specific values are shown in table 5. In general, the corrosion potential characterizes the difficulty of opening corrosion. The higher the corrosion potential, the more difficult or slower the opening of the corrosion process is. Therefore, from the corrosion potential data, the 1.0%N surfacing layer has better corrosion resistance. For the corrosion current, as the nitrogen content increases, the corrosion current shows a decreasing trend. When the nitrogen content is 1.0%, the corrosion current of the surfacing layer reaches the minimum value, which is 1.569 × 10⁻⁶ mA/cm². The self-corrosion current density decreases with the increase of the potential, and the trend is basically the same, and there are obvious passivation areas. The width of the passivation zone is basically the same. For 0.2% N, a passivation zone appears at −0.6V. With the gradual increase of the point, the passivation effect disappears at about 0.3V, and the corrosion current density increases at a fixed rate of increase. Therefore, the passivation range is −0.6V to 0.3V, the passivation current density is about 0.5 × 10⁻⁴ mA cm⁻², the pitting potential is 0.3V, and the passivation current density is the largest. When the amount of nitrogen added is 1.0%, the passivation range of the surfacing layer is −0.8V to 0V, the passivation current density is about

![Figure 5. Polarization curve of different nitrogen content.](image)

| Sample  | Corrosion potential(V) | Corrosion current(mA·cm⁻²) |
|---------|------------------------|----------------------------|
| 0.2%N   | −0.138                 | 2.551E-4                   |
| 0.4%N   | −0.184                 | 1.969E-4                   |
| 0.6%N   | −0.193                 | 1.027E-4                   |
| 0.8%N   | −0.203                 | 6.346E-6                   |
| 1.0%N   | −0.318                 | 1.569E-6                   |
0.5 × 10^{-5} \text{ mA cm}^{-2}, and the pitting potential is 0V. Comparing the data, it can be found that when the nitrogen addition amount is 1.0%, the passivation current density is the smallest, but the pitting voltage is higher, and the stable passivation current density is much greater than the other four groups. Comparing the electrochemical corrosion of laser arc composite surfacing layer of high nitrogen steel made by Rui Wei, by adjusting the welding process parameters, the corrosion current density of high nitrogen steel is 1.453 × 10^{-3} \text{ mA/cm}^2 under the optimal conditions [14]. In this paper, when 1.0% N element is added, the corrosion current density is 1.569 × 10^{-6} \text{ mA cm}^{-2}, and the corrosion resistance is better than the result. These show that the corrosion resistance of the 1.0% N surfacing layer is better than others.

Although the electrochemical curve can see the influence of N element on its corrosion resistance, it can not clearly reflect the corrosion depth of deposited metal in different stages. The corrosion depth has a direct impact on the service life of components. In order to more intuitively see the relationship between the addition of N element and corrosion rate, According to Faraday’s law, the amount of metal dissolved in the anode is expressed as follows:

\[ \Delta m = \frac{A It}{nF} \]  
(1)

Where I represents the current intensity, t represents the power on time, then it represents the power on, A represents the atomic weight of the metal, n represents the valence, and F represents the Faraday constant (F = 96500 C/mol). The electrochemical corrosion surface area s is regarded as an anode, so the corrosion current density \( i_{corr} \) can be expressed as:

\[ i_{corr} = \frac{I}{S} \]  
(2)

Therefore, the corrosion rate of the surfacing sample \( \nu \) The relationship with corrosion current density \( i_{corr} \) can be expressed as:

\[ \nu_{corr} = \frac{\Delta m}{St} = \frac{A}{nF} i_{corr} \]  
(3)

Calculate the corrosion rate (mm/year) according to the polarization curve and formula, as shown in the figure 6 below. It can be seen from the figure 6 that when N element is added to 1.0%, the corrosion rate is the smallest, which is confirmed by the conclusion of polarization curve[15].

In order to study the passivation characteristics with different N content on the surfacing layer in 3.5% NaCl solution, the AC impedance experiment is further carried out on the samples, and the electrochemical impedance spectroscopy (EIS) is shown in figure 7. It can be seen from figure 7 that the capacitive reactances of the surfacing layer with different N additions are semi-circular arc shape, which show that the corrosion mechanism of them are the same. As the amount of N gradually increases, the radius of the capacitive arc of the sample becomes larger and larger. In the Nyquist plots, the larger the radius of the capacitive arc, the better the
corrosion resistance of the surfacing layer. These indicate that the corrosion resistance of surfacing layer gradually increases with the addition of N to form a more stable passivation film.

Figure 8 shows the equivalent circuit fitting results of EIS data under different nitrogen additions. Among them, $R_s$ is the electrolyte solution resistance, $CPE_1$ is the electric double layer capacitor, $R_t$ is the charge transfer resistance, $CPE_2$ is the passivation film capacitor, and $R_f$ is the passivation film resistance. The smaller the $R_t$ value, the easier the ions enter the electric double layer. The lower the $R_f$ value, the weaker the passivation film protection. The smaller $CPE_1$ indicates the fewer defects on the surface of the passivation film. The lower $CPE_2$ indicates the stronger the protection of the passivation film [16, 17]. It can be seen from table 6 that the $CPE_1$ and $CPE_2$ values of the surfacing layer with different boron addition amounts are very small, which indicates that the passivation film is very stable. The passivation film resistance $R_f$ is greater than the charge transfer resistance $R_t$, which indicates that the passivation film plays a major role in the corrosion resistance of the material.

Figure 9 shows the Bode diagram of EIS. It can be seen from figure 8 that each curve has a similar trend, which indicates that the pitting reaction mechanism of the sample is the same. With the increase of nitrogen content, the diameter of the semicircle in the bode diagram increases significantly. The absolute impedance value increases at 0.01Hz, indicating that the corrosion of the sample is weakened. When the amount of nitrogen

Table 6. Equivalent circuit fitting results of surfacing layer with different amount of N.

| Sample | $R_s$ (Ωcm²) | $CPE_1$ (Ω⁻¹s⁻¹cm²) | $n_1$ | $R_t$ (Ωcm²) | $CPE_2$ (Ω⁻¹s⁻¹cm²) | $n_2$ | $R_f$ (Ωcm²) |
|--------|--------------|----------------------|-------|--------------|----------------------|-------|--------------|
| 0.2%N  | 5.054        | 1.994E-3             | 0.896 | 6.399E2      | 2.493E-4             | 0.727 | 1.852E3      |
| 0.4%N  | 5.409        | 8.782E-5             | 0.853 | 1.257E3      | 4.887E-4             | 0.723 | 2.758E3      |
| 0.6%N  | 5.760        | 1.016E-4             | 0.845 | 6.303E3      | 3.387E-5             | 1.000 | 4.745E3      |
| 0.8%N  | 5.394        | 5.398E-5             | 0.880 | 7.772E3      | 1.593E-4             | 0.745 | 4.789E3      |
| 1.0%N  | 7.438        | 3.791E-5             | 0.895 | 1.578E4      | 3.464E-5             | 0.818 | 12.438E3     |

Figure 7. EIS of surfacing layer with different N content: Nyquist.
is 1.0%, the phase angle of the surfacing layer is wider in the middle frequency range, and the phase angle is the largest at the low frequency, which indicates that there is a denser passivation film. In the frequency range of 1 Hz to 1 kHz, the impedance curves have a linear relationship with frequency. The change in the impedance curve from 10 kHz to 100 kHz is independent of frequency. At high frequencies, the phase angle is close to 0, which is roughly equal to the solution resistance between the working electrode and SCE.

With the addition of nitrogen, the nickel content is also reduced accordingly, as shown in Table 1. However, the matrix structure of the surfacing metal is still austenite and has not changed, shown as XRD results in Figure 1. This is mainly because nitrogen is also a stabilizing element for austenitization, which can promote the formation of austenite. The stability of the matrix structure is the basis to ensure that the material has good

![Figure 9. Bode diagram of EIS of surfacing layer.](image1)

![Figure 10. Element distribution of 1.0%N surfacing layer.](image2)

![Figure 9. Bode diagram of EIS of surfacing layer.](image1)

![Figure 10. Element distribution of 1.0%N surfacing layer.](image2)
corrosion resistance. In addition, the main reason for the corrosion resistance of this austenitic stainless steel is that it contains a large amount of Cr elements, and these Cr elements and O elements form a Cr₂O₃ dense oxide film to play a protective role. The addition of N element can also form a compound with Cr element. If a large amount of Cr element is consumed in this process, so that the alloy is poor in Cr in a local area, its corrosion resistance will inevitably be greatly reduced.

Figure 10 is the element distribution of 1.0% N surfacing layer. It can be seen from figure 9 that the distribution of elements such as Fe/Ni/Mn/Si/C is relatively uniform. There is no obvious local segregation of these elements. The distribution of Cr, B and N elements has changed, and there is a certain enrichment at the grain boundary position. This shows that there are borides and nitrides at the grain boundary, which is consistent with the previous XRD results and EDS results. For austenitic stainless steels, the precipitation of the second phase at the grain boundary is likely to cause local depletion of chromium at the grain boundary, so that intergranular corrosion is prone to occur, and the corrosion resistance is reduced. From the data in figure 10, there is no chromium depletion phenomenon, and the Cr contents of the 1.0%N surfacing layer are all greater than 13% (shown as table 3). This shows that the addition of N element formed a hard phase precipitate, but does not appear to be poor in Cr, so it does not lead to a decrease in corrosion resistance. This is because when the nitrogen content increases, it can block the nucleation and growth of chromium-rich carbides, which reduces the probability of chromium-depleted areas at the grain boundaries. Increasing the nitrogen content of austenitic stainless steel can increase the activity of chromium in the steel and slow down the migration rate. The segregation ability of nitrogen far exceeds that of carbon, so the nitrogen content in austenitic stainless steel
increases, and it is easier to react with chromium to form nitrides, which in turn hinders the precipitation of carbides. In addition, the chromium required by the combination of carbon and chromium to form carbides far exceeds the chromium required for nitrogen and chromium to form nitrides, so that chromium-depleted areas are not prone to appear at the grain boundaries in austenitic stainless steel [18].

In order to more clearly reflect the distribution morphology of each phase, the 1.0%N surfacing layer is observed by high-power SEM, and the results are shown in figure 11. It can be found from figure 11 that the precipitated phase accumulation zone is not formed by a single precipitated phase, but by many fine precipitated phases dispersed on the austenite matrix. This should be the reason for the uniform distribution of Ni in figure 10. This distribution of phase can effectively reduce element segregation and improve the corrosion resistance of the material. Nitrogen is beneficial to affect the kinetics of the repassivation potential, and can quickly repassivate to inhibit the growth of pitting corrosion. It can be known from the adsorption theory that charged N\(^{5-}\) is segregated to the passivation film [14], and the passivation film adsorbs anions to cause accelerated corrosion. As the reaction proceeds, the passivation film becomes thinner, so that the adsorbed Cl\(^{-}\) and the segregated N\(^{5-}\) are close. When the passivation film is penetrated, Cl\(^{-}\) and N\(^{5-}\) repel in the same direction, and the repulsive reaction causes Cl\(^{-}\) to desorb. This is the reason why the nitrogen-containing steel is repassivated at the pitting corrosion caused by the erosion and movement of anions. At the same time, this is also the reason why nitrogen atoms or nitrogen ions can improve passivation in the oxide film layer. The action and corrosion mechanism of element N are shown in the figure 12.

4. Conclusion

Through the test and analysis of the microstructure and corrosion resistance of surfacing metals with different nitrogen content, the following conclusions are drawn:

1. The matrix structure of the surfacing metal under different nitrogen content is Fe-Ni-Cr austenite. The addition of nitrogen does not change the matrix structure, but with the increase of nitrogen content, the precipitation of nitrides (Cr\(_2\)(C,N) and BN) is accompanied.

2. When the amount of nitrogen added is 1.0%, the corrosion resistance of the surfacing metal are the best. At this time, the self-corrosion current density is ca. 1.569 × 10\(^{-6}\) mA cm\(^{-2}\) and the self-corrosion potential is ca. −0.318 V.

3. The addition of nitrogen can promote the formation of austenite and nitride, inhibit the appearance of carbides, avoid the phenomenon of poor Cr, and improve the corrosion resistance of the material.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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