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**PAPER**

**Enhanced strength and corrosion resistance in as-cast TA10 alloys via interstitial carbon solute**

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**Abstract**

The addition of interstitial elements generally has a significant impact on the macroscopic performances of structural alloys. Here, we report a systematic study on the microstructure, corrosion behaviors and mechanical properties of a series of as-cast Ti-0.3Mo-0.8Ni-xC (x = 0.09, 0.12 and 0.15 wt.%) alloys. A typical basket-weave microstructure appears in all specimens, characterized by the lamellar α−phase and intergranular retained β-phase without TiC precipitates within the prior β grain boundary. Based on the electrochemical and immersion tests, doping with C element can significantly improve the corrosion resistance of TA10 alloys; with increasing the carbon content, the passivation current density decreases from 5.750 to 4.938 μA·cm⁻², the breakdown potential increases from −1.115 to −1.223 V versus SCE, the corrosion rate reduces from 15 mm/a to 7.5 mm/a. Moreover, the mechanical properties of TA10 alloys, such as the strength and hardness, can also be enhanced via C alloying. Our results indicate that the addition of interstitial elements should be a potential effective mean to accommodate the comprehensive performances of TA10 alloys.

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**1. Introduction**

Ti alloys are known for their excellent corrosion resistance in a wide variety of environments, due to the spontaneous formation of a stable protective film which can protect substrates from corrosion [1–3]. Therefore, Ti alloys are used in harsh environments such as ships, oil extraction and deep-sea exploration equipment [1, 2]. The commercial Ti-0.3Mo-0.8Ni (TA10) alloys, which consists only of trace amounts Molybdenum (Mo) and Nickel (Ni), has attracted much attention due to their low-cost and excellent processing capability; however, the comprehensive properties of these alloys, especially the corrosion resistant and strength, need to be further improved.

Alloying is one of the main methods to improve the corrosion property of titanium alloy [3–6]. For instance, Xia et al [7] instantiated the influence of Zr (0, 10, 20, 30, 50 wt.%) content on the corrosion performance of Ti-6Al-4V alloy and revealed that the passivation ability of Ti-6Al-4V-xZr alloy increases with increasing Zr content. However, excess Zr content may reduce the content of ZrTiO₄ phase on metal substrate, causing the Ti-6Al-4V-xV alloy more susceptible to pitting. In a similar vein, Xu et al [8] reported that Ti-xMo shows an increased corrosion resistance with a higher Mo content, making the Ti-16Mo alloy a promising candidate for orthopaedic applications. Accordingly, the alloying is an expedient and practical way to improve the comprehensive properties of Ti alloys.

What’s more, it has been reported that the carbon, an important element, plays a vital role in enhancing the comprehensive of metals. In this regard, Ogden et al [9] reported the effect of three interstitial solutes (nitrogen, oxygen, and carbon) on the mechanical properties of high-purity alpha titanium, and found that carbon can

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improve the mechanical properties. Carbon can affect the passivation film on the metal surface to improve the corrosion performance. Chiba et al. [10] studied the corrosion resistance of Fe-33Mn-xC austenitic steel and clarified that interstitial carbon can enhance the electrochemical property of the steel. For instance, Luo et al. [11] reported that the addition of 0.5 at. % carbon and Cr and Co endows the interstitial equiatomic CoCrFeMnNi high-entropy (HEAs) with the best pitting resistance. In addition, Su et al. [12] studied the effect of carbon on the corrosion properties of titanium alloys and illustrated that addition of carbon can effectively alleviate the corrosion degree of prior-β grain boundaries. Based on the above analysis, trace carbon can improve the comprehensive properties (mechanical properties and corrosion).

Moreover, it should be noted that the introduction of carbon easily leads to the formation of TiC\(_x\) phase, which can result in the increase of the critical stress for slip. Hosoda et al. [13] reported that the effect of carbon (0.2 ~ 0.5 wt. %) on TiNb alloy and found the grain size decreased with increasing carbon content because of the grain boundary pinning effect of the resulting TiC\(_\beta\) particles. However, adding this level of carbon as an alloy element also generally risks embrittlement of the alloy due to carbon formation. In the work, we try to improve the corrosion performance and the mechanical properties of titanium alloys by adding trace carbon (0 ~ 0.15 wt.%).

In this regard, we select interstitial C element to optimize the comprehensive properties of TA10 alloys. The potentiodynamic polarization (PDP), electrochemical impedance spectroscopy (EIS), and immersion tests have been employed to analysis the corrosion behaviors of Ti-0.3Mo-0.8Ni-xC (x = 0.09, 0.12 and 0.15 wt.%) alloys. The effects of C doping on the microstructure and mechanical properties of TA10 have also been investigated. Finally, we discuss the underlying mechanism about the appearance of C-induced anti-corrosion and strengthen for TA10 alloys.

2. Experimental methods

2.1. Materials

Ti-0.3Mo-0.8Ni-xC alloys (x = 0, 0.09, 0.12 and 0.15 wt.%, denoted as TA10, 0.09 C, 0.12 C, 0.15 C, respectively) were prepared by non-consumable vacuum arc melting commercial sponge Ti (99.9 wt.%), Mo (99.9 wt.%), Ni (99.9 wt.%) and C (99.9 wt.%). In order to ensure uniform alloy composition, every ingot was completely remelted at least six times in this work. The measured chemical composition of these alloys is listed in table 1. The ingots were processed into 10 × 10 × 5 mm\(^3\) squares as experimental specimens for the subsequent tests.

| Alloy no. | Ti   | Mo  | Ni  | C       |
|----------|------|-----|-----|---------|
| TA10     | Bal. | 0.28 ± 0.06 | 0.85 ± 0.08 | —       |
| 0.09 C   | Bal. | 0.31 ± 0.04 | 0.76 ± 0.05 | 0.09 ± 0.01 |
| 0.12 C   | Bal. | 0.29 ± 0.05 | 0.79 ± 0.06 | 0.11 ± 0.02 |
| 0.15 C   | Bal. | 0.29 ± 0.02 | 0.81 ± 0.03 | 0.15 ± 0.01 |

2.2. Microstructure characterization

Microstructure was respectively characterized by an optical microscope (OM, Olympus-GX71, Japan), a JXA-8230 microscope (JEOL Japan) and a Talos F200X microscope (FEI, USA). X-ray diffraction (XRD, Empyrean, Panalytical, Netherlands) was employed (with Bragg-Brentano geometry and Cu Ka radiation) to determine the phase compositions. Corrosion surface morphology of the specimens were observed by Atom Force Microscopy (AFM), after immersing for one hour. The specimens were firstly grinded with SiC papers from 400-grit to 2000-grit, followed by mechanical polishing with 1 μm diamond paste. Then, the samples were etched using Kroll’s reagent of 3 ml HF, 9 ml HNO\(_3\), and 24 ml H\(_2\)O for 10–13 s. Finally, all specimens were cleaned with alcohol and acetone.

2.3. Electrochemical measurements and static immersion tests

Electrochemical measurements were conducted using a standard three-electrode system electrochemical workstation (CHI660E; shanghai chenhua instrument co, LTD, China) in 5 M HCl solution at 25 ± 1 °C (RT) maintained by a thermostatic water bath. In this work, a saturated calomel reference electrode (SCE) and a platinum plate counter electrode (CE) were adapted with the specimen working electrode (WE) to perform the electrochemical tests. All specimens were embedded in epoxy resin, leaving an exposed surface area of 1 cm\(^2\).
Potentiodynamic polarization was conducted from −750 mV to 2500 mV versus SCE with a slow potential sweep rate of 0.001 mV s⁻¹. EIS measurements were performed in a frequency range from 10 mHz to 100 kHz using a sinusoidal potential perturbation of 10 mV. Before the potentiodynamic polarization and EIS measurements, the Open Circuit Potential (OCP) was measured for 7000 s to obtain a reasonably stable potential value. Besides, Zsimpwin software was used to fit the EIS data. All the measurements were repeated at least three times to check the repeatability.

Static immersion tests were carried out in accordance with ASTM Standard G31–72 [14]. The samples were ground to 400-grit with SiC paper and then immersed in 5 M HCl solution at 25 °C using a thermostatic water bath for 10 d. The specimens were taken out from corrosion solution after 2 d immersion, cleaned by alcohol.
and distilled water for 8 min, dried, weighted and finally put in fresh immersion solution again, which was reported by Su et al [15]. The average corrosion rate (CR) was calculated using following formula [16]:

$$CR = \frac{8.76 \times 10^4 \times \Delta w}{S \times t \times \rho}$$

Where, CR is the average corrosion rate in mm/a, Δw is the weight loss in g after 240 h of immersion; S is the exposed surface area of the specimens in cm²; t is the immersion time in hour (h); ρ is the density of each alloy in g/cm³ measured by automatic density measuring instrument (MH-300A). For the same alloy, three specimens were tested to ensure the reproducibility and an average value was reported.

2.4. Mechanical tests
The compression tests were carried out on electronic universal testing machine (Instron 5569, USA) with a loading rate 9 μm s⁻¹ and a strain rate of 1 × 10⁻³ s⁻¹. The samples with the size of Φ 3 mm × 5 mm was tested at RT. The elastic modules were determined by fitting slope of elastic stage of compressive curve. The room-temperature hardness of materials was tested by an HVS-50 digital Vickers hardness tester. A constant load is 1.961 N (200 gf). We measured ten points for four samples, removed the maximum and minimum values and calculated the average value of the remaining data as the final hardness of the material.

3. Results and discussions
3.1. Effect of carbon content on microstructure
The XRD pattern of the Ti-0.3Mo-0.8Ni-xC (x = 0, 0.09, 0.12, 0.15 wt.%) alloys are displayed in figure 1(a). As shown in figure 1(a), the peaks of all alloys can be identified as main α-Ti phase with hexagonal close-packed (hcp) and trace β-Ti phase with body-centered cubic (bcc). With further increasing the C content to 0.15 wt.%, the peak intensity of α (101) and β (110) obviously ascends depicted in figure 1(b). Meanwhile, in figure 1(b), the peak position of the α (101) and β (110) of four titanium alloy shifted slightly toward high angle side compared to the standard peak position, implying a decreased lattice parameter in accordance to the Bragg equation.

Micrographs of the as-cast Ti-0.3Mo-0.8Ni-xC alloy are mapped in figures 2(a)–(h). As presented in figures 2(a), (d), the microstructure of the as-cast TA10 alloy which differs from three other carbon-based titanium alloys is characterized by the lamellar structure known as Widmanstatten structure. It can be inferred that the introduction of carbon not only transform microstructure of the as-cast TA10 alloy from Widmanstatten structure to basketweave microstructure, but also the microstructure of titanium alloy became denser with the increase of carbon content from the figures 2(b)–(d). Furthermore, to clearly identify the retained β-Ti phase, we carried out the SEM experiments in figures 2(e)–(h). The bright areas represented retained the β-Ti phase, while the dark areas corresponded to the α-Ti phase. With increasing carbon content, the amount of the β-Ti phase significantly boosted, in accordance with the result of the OM analysis.

Figures 2(i)–(j) illuminates element distribution of the TA10 and 0.15 C alloy in different phase by TEM. As depicted, it was obvious that Ni element was abundant in β-Ti phase, meanwhile Mo served as an easily
passivated element which can decrease anodic activity of titanium alloy was more inclined to be distributed in β-Ti phase. There was no significant difference in the distribution of C element, indicating that C was an interstitial element in this work.

3.2. Effect of carbon content on corrosion behavior

3.2.1. PDP tests

To further clarify the influences of C additions on the corrosion performance of the Ti-0.3Mo-0.8Ni-xC alloys, the PDP tests were employed at RT, after the OCP reached a stable value. All alloys exhibit similar polarization behaviors during the measurements, as presented in figure 3. The cathodic curves exhibit linear tafel characteristics, implying the main reaction is hydrogen evolution reaction (HER) in 5 M HCl solution [17]. Apparent passivation zone can be observed in four titanium alloy without the evident transition from activation to passivation for the anodic curves. At even higher voltages we enter the dielectric breakdown region of the passive layer, where oxygen is now evolved on the oxide surface [18]. The transition current was caused by the oxygen evolution reaction, and further disappearance occurred due to increased resistivity of the oxide [19].

Meanwhile, we fitted some important parameters of the polarization curve and listed in table 2, such as corrosion potential, corrosion current density, breakdown potential and passivation current density. The decreased corrosion potential and the increased corrosion current density which was obtained by the Tafel extrapolation method illuminated that the corrosion resistance of TA10 was improved with further increasing carbon content. In this work, the data of breakdown potential and passivation current density which were more targeted in the passivation system were mapped in figure 4. The passivation current gradually decrease and the breakdown potential showed the opposite trend. Therefore, the resistance to corrosion of the Ti-0.3Mo-0.8Ni-xC alloys can be put in the following descending order: 0.15 C > 0.12 C > 0.09 C > TA10. The barrier-type oxide film would be formed on the whole of the specimens, which was indicated by the extremely low passive current densities [7]. Besides, in the PDP curves, the breakdown potential is the most significant parameters for evaluating the breakdown corrosion behaviors [20]. The breakdown potential of the TA10 alloy was the lowest at 1.15 ± 0.02 V versus SCE manifesting that the addition carbon can improve the breakdown resistance of the TA10 alloy.

Table 2. Parameters deduced from potentiodynamic polarization curves.

|                  | $E_{corr}$ (V versus SCE) | $i_{corr}$ ($\times 10^{-2}$μA·cm$^{-2}$) | $i_{pass}$ (μA·cm$^{-2}$) | $E_b$ (V versus SCE) |
|------------------|--------------------------|------------------------------------------|----------------------------|-------------------|
| TA10             | −0.364 ± 0.012           | 9.937 ± 0.15                             | 5.750 ± 0.006              | 1.115 ± 0.008     |
| 0.09 C           | −0.327 ± 0.010           | 8.556 ± 0.12                             | 5.164 ± 0.009              | 1.134 ± 0.013     |
| 0.12 C           | −0.204 ± 0.007           | 7.326 ± 0.09                             | 5.056 ± 0.005              | 1.153 ± 0.014     |
| 0.15 C           | −0.164 ± 0.018           | 7.064 ± 0.23                             | 4.938 ± 0.012              | 1.223 ± 0.013     |

Figure 4. Variation of experimental breakdown potential and passive current density with C contents for the Ti-0.3Mo-0.8Ni-xC (x = 0, 0.09, 0.12 and 0.15 wt. %) alloys in 5 M HCl.
3.2.2. EIS measurements

To evaluate the electrochemical properties of the formed passivation film with the various carbon content, EIS measurements were performed. Figure 5 presents the corresponding Nyquist and Bode plots for the Ti-0.3Mo-0.8Ni-xC alloy in 5 M HCl solution. It can be observed from the Nyquist plots that there exist two capacitive loops for the Ti-0.3Mo-0.8Ni-xC alloy, which is generally indicative of two capacitive time constant. One at the medium and high frequency which is attributed to the charge transfer process of Ti/Ti^4+ at the double layer formed at the samples surface and the other at the low frequency which is closed to impedance produced by oxide or porous corrosion product film [21–23]. The diameter of the first capacitive loops is equal to the charge transfer resistance of the samples [24, 25]. As shown in figure 5(a), the radius of the capacitive loops increases gradually with increasing the carbon content, indicated the increasing corrosion resistance. There was no obviously difference between the 0.12 C and 0.15 C alloy in high frequency, whereas, the diameter of the 0.15 C alloy is bigger than that of the 0.12 C alloy in low frequency (0.01–0.1 Hz), indicating the charge transfer for the 0.15 C alloy is enormously limited. In the Bode plots mapped in figure 5(b), the phase angles approaching 80° in the medium frequencies can be seen in the case of 0.09 C, 0.12 C, 0.15 C, while the peak value of phase angle of TA10 was in approximately 65°. Meanwhile, the Bode magnitude of the as-cast Ti-0.3Mo-0.8Ni-xC alloys increases with the carbon addition. Hence, these facts indicate that carbon can effectively increase the corrosion resistance of the Ti-0.3Mo-0.8Ni-xC alloys.

In order to better elucidate the corrosion behaviors of the Ti-0.3Mo-0.8Ni-xC alloy, the R(Q(R(Q))) equivalent circuit with two-time constants which is an indication of a double layer film, mapped in figure 6 [5, 26, 27] was used to fit the EIS data. In the equivalent circuit, the Rs is the electrolyte resistance, Rf and Qf represent the film resistance and its corresponding constant phase element, respectively, and Rct and Qdl indicate the charge transfer resistance and the constant phase element of the electrical double-layer at the sample surface. The non-ideal behavior of non-uniform interface is characterized by a constant-phase elements (CPE) in the work [28], and its impedance (Z_{CPE}) can be defined by the equation following:

![Figure 5. Electrochemical impedances spectra of the Ti-0.3Mo-0.8Ni-xC (x = 0, 0.09, 0.12 and 0.15 wt. %) alloys in 5 M HCl solution: (a) Nyquist plots, (b) Bode plots. Symbols represent the experimental data and lines represent the fitted data obtained by Zsimpwin 3.10 software.]

![Figure 6. The equivalent circuit used to fit the measured EIS data of the Ti-0.3Mo-0.8Ni-xC (x = 0, 0.09, 0.12 and 0.15 wt. %) alloys.]

Where, $Q$ is the CPE constant (the equivalent of the system’s admittance at 1 rad s$^{-1}$), $j$ is the imaginary root, $\omega$ is the angular frequency, and $n$ is the deviation parameter ranging from $-1$ to 1.

Table 3 displays the fitting results of the Nyquist and Bode plots. The chi-square ($\chi^2$) values are in the order of $10^{-3}$, demonstrating an excellent fitting quality. Generally, polarization resistance ($R_p$) can be adopted to evaluate the corrosion resistance. The $R_p$ can be expressed as follows: $R_p = R_{ct} + R_{ct}$. As listed in table 3, the $R_p$ of the TA10 alloy is the minimum value at 343.6 $\Omega \cdot \text{cm}^2$, illuminating that its corrosion resistance is also terrible. The $R_p$ for 0.09 C, 0.12 C and 0.15 C is equal to 993.5 $\Omega \cdot \text{cm}^2$, 1222.7 $\Omega \cdot \text{cm}^2$ and 1923.7 $\Omega \cdot \text{cm}^2$, respectively. The improved corrosion performance of Ti-0.3Mo-0.8Ni-xC alloys by C alloy can be contributed to the difference of composition and microstructure.

3.2.3. Static immersion tests
The static immersion tests were conducted to illuminate intuitively the corrosion resistance of the Ti-0.3Mo-0.8Ni-xC alloys. Figure 7 depicted the results of the weight loss of the Ti-0.3Mo-0.8Ni-xC alloys after 10 d of immersion. The weight loss gradually decreases for each immersion time with increasing the carbon contents. Meanwhile, the weight loss of the 0.15 C alloy is approximately 75 mg cm$^{-2}$, a half of TA10 alloy, manifesting directly that carbon addition improves the corrosion resistance of the TA10 alloy. The difference for weight loss can be attributed to the varieties of chemical composition and microstructure. The results of the immersion tests are accordance with results of the PDP and EIS measurements. Besides, according to the weight loss curves mapped in figure 7(a), the corrosion rate of Ti-0.3Mo-0.8Ni-xC alloys was calculated by the formula (1), as depicted in figure 7(b). With increasing the carbon content from 0 to 0.15 wt.%, the corrosion rates decreased from 15 to 7.5 mm/a, which illuminates that the carbon plays a significant role in improving the corrosion resistance of titanium alloys.

In order to further study the corrosion resistance of $\alpha$ and $\beta$ phase, AFM experiments for the TA10 and 0.12 C alloys were employed after immersion in 5 M HCl for one hour at RT. Figure 8 represents the corresponding results of corroded surfaces. Based on the analysis of the HADDF and SEM (figure 2), the relative positions of $\alpha$ and $\beta$ phase on corroded surface of the TA10 and 0.12 C alloys are marked in figures 8(a)–(d). As demonstrated figures 8(e), (f), the relative height of $\beta$ phase is higher than $\alpha$ phase which directly shows that $\alpha$
phase is preferentially dissolved and the corrosion resistance of β phase is better than α phase under same experimental conditions. This result provides favorable support for the formation reason of corroded surface morphology.

3.3. Effect of carbon content on mechanical properties
Figure 9(a) shows the compressive true stress-strain curves of the as-cast Ti-0.3Mo-0.8Ni-xC alloys. The compressive curves of the 0.09 C, 0.12 C and 0.15 C alloys are very similar. During the compression tests process, only the TA10 alloy did not break, illuminating this alloy has the best ductility. However, the 0.09 C, 0.12 C and 0.15 C are sheared fractured along 45° of the axis. Mechanical parameters of the Ti-0.3Mo-0.8Ni-xC alloys such as Young’s modulus, yield strength, compressive strength and plastic strain are listed in table 4. As depicted table 4, compared with the TA10 alloy, the Young’s modulus and yield stress are improved due to the introduction of carbon indicating that the carbon addition can increase the Young’s modulus [29]. The increased strength is mainly attributed to the content of interstitial carbon element. On the one hand, carbon could cause lattice distortion and efficaciously pin the dislocations to provide a solid solution strengthening effect. On the other hand, the interstitial carbon atom in the octahedral site of Ti lattice can purpose a tremendous increase of charge density around them, and further lead to remarkable elevation in the theoretic strength [30]. There is so little difference of compressive strength among 0.09 C, 0.12 C and 0.15 C. Besides, the introduction of carbon makes the plastic strain substantial decrease, which can be attributed to strengthening effect spawned by the addition of carbon [31].
The microhardness of the Ti-0.3Mo-0.8Ni-xC alloys is shown in figure 9(b). It’s intuitive that the microhardness of the TA10 alloy is enhanced by adding carbon from 195.4 HV to 213 HV in line with the result from previous studies [9, 32–34]. The reason can be attributed to the difference of alloy elements and structure which is more refined due to carbon addition.

### 4. Conclusions

In this work, the microstructure, corrosion behavior and mechanical properties of the Ti-0.3Mo-0.8Ni-xC (x = 0, 0.09, 0.12 and 0.15 wt.%) alloys are investigated. The main results are as follows:

1. Microstructure studies illuminate that the microstructure changes from Widmanstatten structure to basketweave microstructure which is much finer. Meanwhile, the amount of β-Ti phase increases with increasing carbon content.

2. The corrosion resistance of the Ti-0.3Mo-0.8Ni-xC alloys is improved with carbon addition confirmed by static immersion and electrochemical tests. There is the galvanic corrosion between α-Ti phase and β-Ti phase on the corroded surface and the corrosion performance of β-Ti phase is better than α-Ti by AFM tests.

3. In the mechanical tests, the microhardness of the Ti-0.3Mo-0.8Ni-xC alloys are lightly enhanced with carbon addition. The introduction of carbon makes the TA10 alloy fracture. The plastic strain of the titanium with carbon content increase from 40.39% to 44.84%. Besides, Young’s modulus and yield stress obviously increase when the carbon content is in the range from 0.09 to 0.15 wt. %.

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Data availability statement

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

Competing interests

The authors declare no competing interests.

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