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Effect of Iron (Fe) on the Corrosion Performance of NiCr-based Alloys

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INTRODUCTION & AIM

Critical petrochemical industry plant equipment exposed to corrosive chemicals remains susceptible to metal dusting (MD) corrosion, resulting in infrastructure degradation, significant financial losses and huge safety risks. Metal coating is one of the approaches pursued to inhibit metal dusting. Firstly, by the addition of elements inhibiting the catalytic activity of iron (Fe) and nickel (Ni), such as tin (Sn) or copper (Cu). Secondly, high amounts of chromium (Cr), aluminium (Al) and silicon (Si) can be added to establish a stable protective oxide scale. NiCr-based alloys are well known multicomponent alloys with a carefully balanced composition to provide the desired properties for various industrial applications. Although these alloys can resist corrosion to a certain extent, MD can still occur under severe conditions, resulting in serious damage to a system in operation or cause frequent operational downtime, which results in loss of production time and high maintenance cost. The composition of nickel-based alloys can be tailored to specific operating conditions, to improve its reliability [1]. This stems from the fact that alloying Ni-Cr alloy with transition elements such as molybdenum (Mo), iron and copper can improve the corrosion resistance by promoting the oxide formation process which contribute to oxide passivity.

This study is focused on investigating the effect of adding small amounts of Fe to austenitic Ni-Cr-based alloy in the process of developing a coating material that can form stable protective phases on the surface when reacting with the process environment. As the initial development step, the current work reports the results obtained during low temperature corrosion simulation tests (electrochemical potentiodynamic), with the intent to expose the coating alloys to metal dusting environment in the near future.

The alloy compositions considered in this study were produced using arc-melting furnace operating under inert atmosphere to produce metal ingots. The samples were sectioned, followed by subsequent heat treatment in a tube furnace. Phase and microstructural analyses were conducted using X-ray diffractometer (XRD) and optical microscopy (OM), respectively. Using electrochemical potentiostat, corrosion resistance trend at various Fe content was established.

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METHOD

- Arc-melting furnace was used to produce the coating alloys, followed by subsequent heat treatment in a tube furnace. Phase and microstructural analyses were conducted using X-ray diffractometer (XRD) and optical microscopy (OM), respectively.
- Electrochemical corrosion tests were carried out using potentiodynamic tests undertaken according to the ASTM Standard (ASTMG5, 2013) [2]. A PC-driven ACM Instruments AutoTafel potentiostat was used to perform the electrochemical tests. One graphite rod acted as counter-electrodes and a Haber-Luggin capillary made the junction with a saturated calomel reference electrode (SCE). All potential values were measured with respect to the SCE. The test was performed at 65±1°C in 5% NaCl + 0.5% acetic acid solution while stirring at 80rpm with a magnetic stirrer. Nitrogen was bubbled continuously during testing to remove all the oxygen and maintain an anaerobic condition. After each sample was immersed in the solution, the open circuit potential (OCP) against time curve was recorded for up to 1 hour. When the potential had reached a sufficiently stable value at 1 hours, a cyclic polarisation scan was recorded from –350 mV to 1500 mV at a scanning speed of 10 mV/min. The scan direction was not reversed. The corrosion rates were then calculated using Equation 1 below:

$$C_R = \frac{I_{Corr} \cdot K \cdot E_W}{D \cdot A} \qquad [1]$$

where: C_R = The corrosion rates, Icorr = The corrosion current in amperes

K = A constant that defines the units for the corrosion rate , E_W = The equivalent weight in grams/equivalent
D = Density in grams/cm³, A = Area of the sample in cm²

Figure 1: a) shows the photographs of the Amazemate melting machine, b) the Tube furnace and c) the electrochemical test cell.

RESULTS & DISCUSSION

Corrosion – Electrochemical tests

Table 1: Potentiodynamic polarisation corrosion results of the samples.

Alloy	E_{Corr} (mV SCE)	I_{corr} (mA/cm²)	Corrosion Rates (mm/y)
1WQ - NiCrMoWFe$_0$	-316±21	3.37x10^{-3}±0.002	0.037±0.016
3WQ - NiCrMoWFe$_1$	-344±16	3.37x10^{-4}±0.005	0.0037±0.007
5WQ – NiCrMoWFe$_3$	-266±55	6.61x10^{-4}±0.000	0.0073±0.002

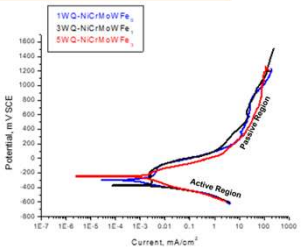

Figure 2: Potentiodynamic polarisation curves of the tested samples.

- The potentiodynamic polarisation corrosion results of the tested samples are shown in Figure 2. The potentiodynamic polarisation corrosion results of the tested samples are shown in Figure 2. According to Fontana [3], the corrosion performance of 1WQ - NiCrMoWFe0 alloy can be classified as excellent because the corrosion rate was 0.04 mm/ y which is in the range of 0.02 – 0.1 mm/y according to Fontana criterion. 3WQ - NiCrMoWFe1 & 5WQ – NiCrMoWFe3 sample can be classified as exhibiting outstanding corrosion resistance of 0.004 & 0.007mm/y which falls in the range of <0.02 mm/y according to Fontana criterion. When NiCrMoW was added with Fe, the corrosion resistance increased significantly. This behaviour might be attributed to the smaller amounts of Fe added although the corrosion rate for 5WQ – NiCrMoWFe3 is higher than that of 3WQ - NiCrMoWFe1. It is important to note that high Fe addition might be detrimental to the alloys [3].

Metallography

- The samples were ground and polished to 1µm and etched with a mixture of $FeCl_3$ and HCl solution to reveal the microstructures. A typical microstructure of a NiCrMoW alloy usually consists of a single γ phase face-centered cubic (FCC) solid-solution nickel-rich matrix with refractory-rich precipitates (due the presence of tungsten and molybdenum). When 1% of Fe was added to the NiCrMoW alloy the grains became more pronounced and refined and the corrosion rate increased. The same can be said about the addition of 3% Fe to the NiCrMoW alloy.

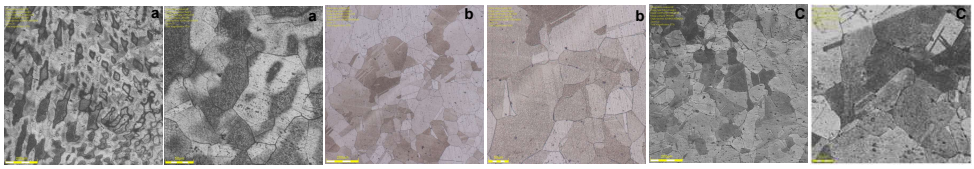

Figure 3: Micrographs of (a) NiCiMoWFe0 – WQ showing nickel-rich matrix with refractory-rich precipitates (tungsten and molybdenum carbides , b) NiCiMoWFe1 - WQ & c) NiCiMoWFe3 - WQ shows more refined grains.

Scanning Electron Microscopy

- The alloy compositions were confirmed with SEM-EDS technique revealing that there is no segregation or formation of intermetallic phase, resulting in only γ-single phase matrix with minor metal oxide inclusions.

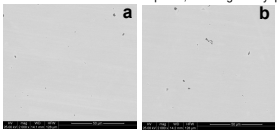

Figure 4: SEM micrographs of the considered NiCrMoWFe$_3$ alloys in the (a) furnace cooled (FC) and (b) water quenched (WQ) conditions upon soaking at 1100 °C.

X-Ray Diffraction

- The phases were identified using X'Pert Highscore plus software. The phases present in the samples were detected through X-ray diffraction (XRD) analysis. With different iron contents, diffraction peaks corresponding to the face-centered cubic (FCC) γ-Ni crystal planes (111), (110), and (211) are evident. This observation suggests that the phase present in the Ni-Cr-Mo-xFe alloy primarily consists of the FCC γ-Ni solid solution. it is well established that the introduction of larger-radius atoms (Cr, Mo, Fe) into the Ni lattice for solid solution formation results in lattice expansion and distortion. Consequently, this expansion leads to an increase in crystal plane spacing (d) and a reduction in the diffraction angle (θ), causing a shift of the diffraction peak towards lower angles [4].

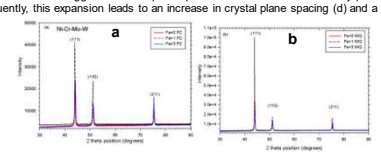

Figure 5: XRD patterns of the considered Ni alloys in the (a) furnace cooled (FC) and (b) water quenched (WQ) conditions upon soaking at 1100 °C.

CONCLUSIONS

- An improvement in the corrosion resistance was shown when Fe was added to the NiCrMoW alloy. Although the corrosion rare for 5WQ – NiCrMoWFe3 is higher than that of 3WQ - NiCrMoWFe1.
- A typical microstructure of a NiCrMoW alloy usually consists of a single γ phase face-centered cubic (FCC) solid-solution nickel-rich matrix.
- The alloy compositions were confirmed with SEM-EDS technique revealing that there is no segregation or formation of intermetallic phase, resulting in only γ-single phase matrix with minor metal oxide inclusions.
- With different amount of Fe addition, diffraction peaks heights corresponding to the FCC γ-Ni crystal planes (111), (110), and (211) are evident, maintaining single phase.

FUTURE WORK/ REFERENCES/ACKNOWLEDGMENT

FUTURE WORK: Current work reports the results of the initial plan of undertaking low temperature corrosion simulation tests (electrochemical), with the intent to expose the coating alloys to metal dusting environment, which is high temperature, in the near future.

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