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Hot corrosion behavior of CoCrFeNiAl HEA produced by powder metallurgy in NaCl molten salt

Toz metalurjisi ile üretilen CoCrFeNiAl YEA'nın NaCl erimiş tuz içindeki sıcak korozyon davranışı

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Highlights

- ❖ The hot corrosion behavior of CoCrFeNiAl HEA was explored.
- ❖ The role of NaCl salt in the degradation process was emphasized.
- ❖ The corrosion scale was porous and loose.

Graphical Abstract

In this work, the hot corrosion behavior of CoCrFeNiAl high entropy alloy (HEA) produced by powder metallurgy was investigated.

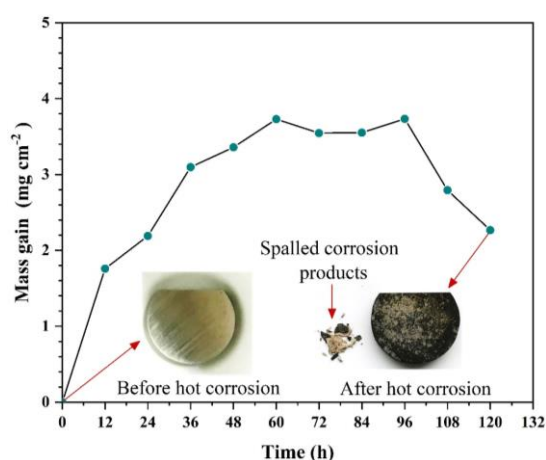


Figure. Mass gain plot for the hot corroded CoCrFeNiAl HEA in NaCl molten salt at 850°C for 120 h

Aim

This work aims to study the hot corrosion behavior of CoCrFeNiAl HEA in NaCl molten salt environment at 850°C for 120 h.

Design & Methodology

CoCrFeNiAl high entropy alloy was obtained by using a powder metallurgy approach. The hot corrosion experiment was performed in a cyclic mode and in a NaCl-containing environment.

Originality

The hot corrosion behavior of CoCrFeNiAl HEA produced by powder metallurgy merged with resistance sintering method has not been addressed in the open literature before. Therefore, the present work can provide insight and database to investigate of hot corrosion behavior of HEAs with various compositions.

Findings

CoCrFeNiAl HEA exposed to NaCl molten salt was subjected to severe hot corrosion attacks, with the result that the corrosion scale could not provide further protection.

Conclusion

The corroded scale formed on the HEA was porous and loose, most probably a consequence of self-sustaining cyclic reactions.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Hot Corrosion Behavior of CoCrFeNiAl HEA Produced by Powder Metallurgy in NaCl Molten Salt

Araştırma Makalesi / Research Article

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ABSTRACT

The present work reports the hot corrosion behavior of equimolar CoCrFeNiAl high entropy alloy (HEA) produced by powder metallurgy. The hot corrosion behavior of the HEA was characterized in severe conditions involving NaCl molten salt environment and cyclic mode. The mass gain of CoCrFeNiAl HEA after being hot corroded at 850°C for 120 h was about 2.2 mg/cm². After 120 h of hot corrosion at 850°C, XRD analysis result revealed that the constituent phases of the scale formed on CoCrFeNiAl HEA were Al₂O₃, Fe₂O₃, Cr₂O₃, NiO and NiCr₂O₄. The corrosion scale mainly consisted of Cr₂O₃, and underneath this layer was dominated by a discontinuous thin layer of Al₂O₃. The role of NaCl on the hot corrosion mechanism was also discussed.

Anahtar Kelimeler: Powder metallurgy, high entropy alloys, CoCrFeNiAl, hot corrosion.

Toz Metalurjisi ile Üretilen CoCrFeNiAl YEA'nın NaCl Erimiş Tuz İçindeki Sıcak Korozyon Davranışı

ÖZ

Mevcut çalışma, toz metalurjisi ile üretilen eşmolar CoCrFeNiAl yüksek entropili alaşımının (YEA) sıcak korozyon davranışını ele almaktadır. YEA'nın sıcak korozyon davranışı, NaCl erimiş tuz ortamı ve döngüsel modu içeren zorlu koşullarda karakterize edilmiştir. CoCrFeNiAl YEA'nın 850°C'de 120 saat sıcak korozyona maruz kalmasının ardından kütle kazancı yaklaşık 2.2 mg/cm² idi. 850°C'de 120 saat sıcak korozyon sonrasında, XRD analiz sonucu CoCrFeNiAl YEA üzerinde oluşan skalayı oluşturan fazların Al₂O₃, Fe₂O₃, Cr₂O₃, NiO ve NiCr₂O₄ olduğunu göstermiştir. Korozyon skalası esas olarak Cr₂O₃'ten oluşuyordu ve bu tabakanın altında, süreksiz ince bir Al₂O₃ tabakası baskındı. Ayrıca NaCl'nin sıcak korozyon mekanizması üzerindeki rolü de tartışılmıştır.

Keywords: Toz metalurjisi, yüksek entropili alaşımlar, CoCrFeNiAl, sıcak korozyon.

1. INTRODUCTION

Dünya'da güneş enerjisi kullanımı yaygınlaşmaktadır. Türkiye'de güneş enerjisinden faydalanma genellikle sıcak su hazırlama şeklindedir. High entropy alloys (HEAs), also termed multi-component alloys, represent a new group of alloy systems comprising multiple metallic elements with different crystal structures at either equiatomic ratio or near equiatomic ratios that can crystallize as simple solid solution phases, including body-centered cubic (BCC) or face-centered cubic (FCC) crystal structures, or both [1,2]. In the recent past, high entropy alloys have received noteworthy attention from both engineers and scientists in the field of materials science thanks to their many outstanding properties including excellent resistance to oxidation and corrosion [3], high strength, and high fracture toughness [4] and good thermal stability [5]. The four core effects of HEAs, i.e., high sluggish diffusion, mixing entropy, cocktail effect and lattice distortion are mainly responsible for the emerging enhanced properties of HEAs [6]. Their unusual performances make them convenient for structural and high-temperature applications, such as

petrochemicals, power aerospace turbines and generation plants [7].

Among all the HEAs, one of the most widely studied HEAs is CoCrFeNiAl metallic system due to its potential for being used in future engineering applications [8]. The casting method has been reported to be the most widely adopted production process for the preparation of HEAs [9]. However, this method can produce unfavorable microstructures with coarse grains, chemical heterogeneity and structural defects such as voids and porosity, resulting in reduced mechanical properties of alloys [10]. Furthermore, the casting process is complex and costly, and it is also hard to control the microstructure, making it an inconvenient method for producing HEAs [11]. Powder metallurgy technology has received great deal of attention from the industrial sectors and material researchers due to the limitations and disadvantages of the casting process [12]. Resistance sintering (RS) process has shown high potential as one of the most important methods for producing a wide variety of engineering materials. The RS, a sintering technology that has emerged as an unconventional technique allows materials with enhanced properties to be readily produced in a short processing time. Moreover, another

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advantage of this method is that it can prevent chemical heterogeneity, provide microstructural control and offer a highly dense microstructure with refined grains. Development of these achieves at fast heating rates and in short sintering durations, without compromising mechanical properties [12-17]. Considering the above considerations, the RS method provides a more innovative and convenient way to produce HEAs compared to other conventional processes. Therefore, in the present work, the resistance sintering method is embraced to produce the CoCrFeNiAl HEA. It is important to highlight that there is a meaningful association between the production process and phase formation. To quote a few examples from previously published studies, the CuCrCoFeNi HEA has been fabricated through casting process by Tong et al. [18] and they found that the constituent phase of the alloy was a single FCC phase. This result was dissimilar to that of Praveen et al. [19] who reported that the same composition of HEA produced by spark plasma sintering (SPS) method was composed of two FCC phases and a minor amount of σ phase.

Hot corrosion can be regarded as an accelerated oxidation attack caused by the condensation of aggressive agents on the surface of a material, with serious consequences in high-temperature applications such as gas turbines, power generation equipment, industrial waste incinerators, and internal combustion engines [13,20]. Hence, it reckons to be crucial to have an understanding hot corrosion behavior of HEAs to meet the requirements of harsh corrosive environments. Moreover, the hot corrosion performance of HEAs in molten salt environments has become a hot spot for new investigations, a large number of works on the HEAs being focused rather on their microstructure characterization, phase formation criteria and mechanical properties. Therefore, the present work was undertaken to investigate the corrosion behavior of CoCrFeNiAl HEA produced by powder metallurgy in NaCl molten salt at 850°C for 120 h.

2. EXPERIMENTAL METHODS

The CoCrFeNiAl HEA was produced in this study by resistance sintering technique starting from elemental powders having purities of higher than 99.9 %. The particle size of elemental powders and milled powders ranged from 5 to 50 μm , i.e. Co (~44 μm), Cr (~5 μm), Fe (~5 μm), Ni (~50 μm), Al (~7 μm) and milled powders (~10 μm). The amount of each constituent element was weighed using an electronic balance with high accuracy of 10^{-5} g as per the equimolar proportion. Equimolar element blends of Co, Cr, Fe, Ni and Al powders were dry milled in a ball milling machine at a speed of 200 rpm with a ball-to-powder weight ratio of 7:1 for 6 h to attain a homogenous powder mixture. The milled powder mass was poured into a steel die with an inside diameter of 20 mm. Subsequently, the steel die was placed between two punches to activate the sintering process. The blended

powder mixture was consolidated using a Resistance sintering (RS) apparatus at the direct electric current of 4 kA, under a uniaxial pressure of 70 MPa and with 20 min holding time.

The corrosion behavior of the resistance sintered HEA was cyclically evaluated in molten NaCl salt at 850°C for 120 h in a muffle furnace. The sample was put in an alumina boat containing NaCl salt, then located in the furnace to render the corrosion experiment. After each cycle of corrosion testing (12 hours), the sample was allowed to cool to laboratory temperature and then it was washed in distilled water at 100°C for 15 min to eliminate residual salts on the surfaces, followed by drying. The same procedure was provided for each corrosion cycle. Assessment of the weight change resulting from the growth and spallation of the formed oxides on the sample was carried out in each cycle of the corrosion test by using a digital balance with an accuracy of 10⁻⁵ g. Moreover, solidworks software was used to determine the surface area of the sample to be hot-corroded.

Following the corrosion experiments, with the aim of characterization of the corroded alloy, the transverse section of the corrosion layer was ground by using SiC papers from P180 to P1200 followed by polished with a 1 μm diamond suspension. The phase constitution of the corrosion scale formed on the sample was investigated on an X-ray diffractometer (Rigaku, D/MAX-B/2200/PC) using Cu K α radiation at a step size of 0.02° and the scanning angle (2θ) was from 20° to 90°. The scanning electron microscopy (SEM, JEOL JSM-6060, LV), and energy-dispersive spectroscopy (EDS) were used to characterize the surface morphology and cross-sectional microstructure of the hot corroded HEA.

3. RESULTS AND DISCUSSION

3.1. Hot corrosion kinetics

Figure 1 displays the corrosion kinetics curve of CoCrFeNiAl HEA exposed to NaCl molten salt at 850°C for 120 h, and macro photos corresponding to before and after hot corrosion of alloy can also be seen in the same Figure. Furthermore, the spalled corrosion products were also collected to manifest the extent of corrosion attack. The data of mass gain per unit surface area with respect to the corrosion time presented in Fig. 1 reflects the hot corrosion behavior of CoCrFeNiAl HEA. The mass gain for each cycle was determined by dividing the weight difference of the HEA weighed before and after exposure with the initial surface area. According to the mass gain graph, the corrosion rate of the HEA increased with increasing exposure time, implying that the increasing time played an essential role in provoking the degradation extent of the alloy. This fact inferred from the graph is in line with the results of the macro photo for the corroded alloy. The kinetic curve of the alloy shows that the hot corrosion process is controlled by three different stages. The first stage corresponded to a rapid mass gain in the initial exposure of 20 h, where the generation of corrosion products on the surface occurred.

Mass gain of the alloy reached about 3.7 mg/cm^2 at the end of this stage without the formation of any scale spallation. The alloy showed a steady stage keeping a minor mass gain in the second stage, which is accompanied by slight spallation of the corrosion product. After exposure of 96 h, which is corresponded to the last stage in hot corrosion process, remarkable mass loss was noted, as can be seen in Fig. 1. The witnessed mass loss provides evidence that the rate of scale spallation is higher than the rate of scale growth. This stage brings about the inferior corrosion resistance in which scale spallation allows direct contact of the molten salt to the surface. After hot corrosion at 850°C for 120 h, the final mass gain data of the alloy was recorded as about 2.2 mg/cm^2 . In addition, the corroded surface of the alloy exhibited a mainly dark gray and yellowish color, and also the protrusions associated with spallation of oxides appeared on the surface. However, in fact, spalled corrosion products caused the appearance of a yellowish color on the scale, as proved by the inset image shown in Fig. 1.

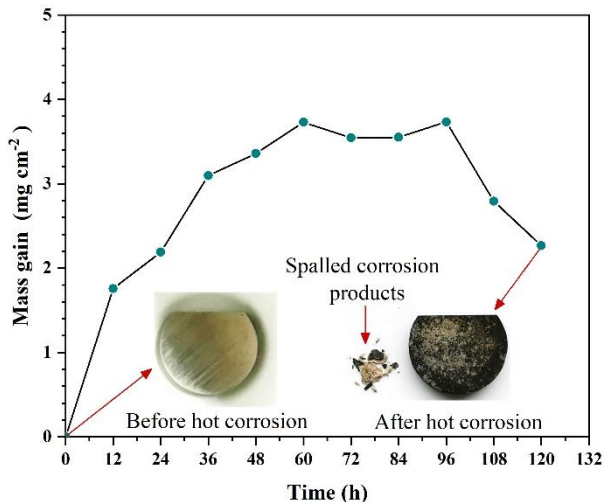


Fig. 1. Mass gain plot for the hot corroded CoCrFeNiAl HEA in NaCl molten salt at 850°C for 120 h

3.2. Characterization of corrosion scale

The phase constitutions of hot corroded CoCrFeNiAl HEA in NaCl molten salt at 850°C for 120 h are examined via X-ray diffraction (XRD), with the analysis result being displayed in Fig. 2. According to the XRD pattern of the hot corroded alloy, the oxides of Al_2O_3 , Fe_2O_3 , Cr_2O_3 , NiO and NiCr_2O_4 were the main corrosion products that formed on the HEA after corrosion at 850°C for 120 h. In addition to these phases, diffraction peaks belonging to an FCC and two BCC structures were also detected. This was most likely due to the fact that a portion of the corrosion products was spalled, and also the scale on the alloy was thin.

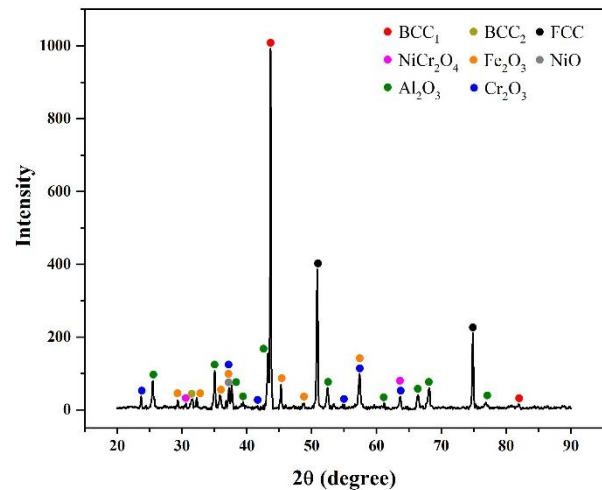


Fig. 2. XRD patterns of the hot corroded CoCrFeNiAl HEA in NaCl molten salt at 850°C for 120 h

Fig. 3 (a-c) corresponds to the surface morphologies of the CoCrFeNiAl HEA after exposure to NaCl molten salt at 850°C for 120 h. Results of the EDS analysis for the analyzed surface are also shown in Fig. 3 (d). The high-magnification micrographs of the corroded surface (Fig. 3 (b,c)) prove that the scale formed on the HEA is mainly characterized by three distinct regions. These regions can be defined as follows: the dark region (marked area with 1), the bright region (marked area with 2) and the gray region (point 3) in Fig. 3 (c). According to the EDS results in Fig. 3 (d), the chemical composition of the dark region (area 1) consisted mainly of Al and O, which encourages the constitution of Al_2O_3 . Whereas the bright region (area 2) on the scale was dominated by Co, Cr, Fe, Ni and O, which was verified through elemental mapping analysis. (Fig. 4). The top layer on the scale (point 3) is enriched with Cr and O, indicating the constitution of Cr_2O_3 , as evidenced by elemental mapping analysis. In addition to these, another region with a relatively small portion (point 4) sitting on the Cr-rich oxide layer is also observed. The EDS result of point 4 shows the existence of Al, Ni, O and Cl in higher concentrations and small amounts of Cr, Co and Fe. The existence of residual Cl on the surface was a result of the corrosion environment. Elemental mapping analysis confirms that the corroded surface is mainly covered by the Cr-rich oxides. Besides, it can be noticed in Fig. 4 that insignificant amounts of Na appear in some regions of the surface. It is worth to note that there was no crack in the corrosion scale on the alloy. However, the spallation of corrosion products was witnessed. This spallation phenomenon can explain the reason for the decrease in mass gain of the alloy during hot corrosion process. Moreover, the fact behind the formation of spallations can be attributed to the highly aggressive corrosion environment and the effect of the cyclic test. Therefore, such a corrosion scale causes direct contact of corrosive molten salt with the substrate so there is a decreased corrosion resistance

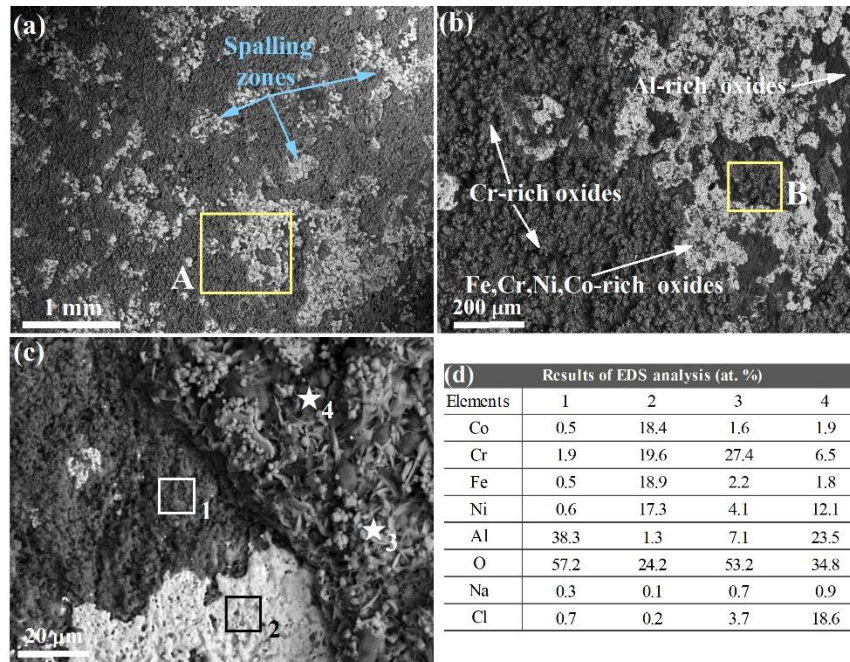


Fig. 3. SEM surface morphologies of the hot corroded CoCrFeNiAl HEA in NaCl molten salt at 850°C for 120 h, (a) low-magnification micrograph of the surface, (b) high-magnification micrograph of the marked area with A, (c) high-magnification micrograph of the marked area with B and (d) EDS results corresponding to points on the surface.

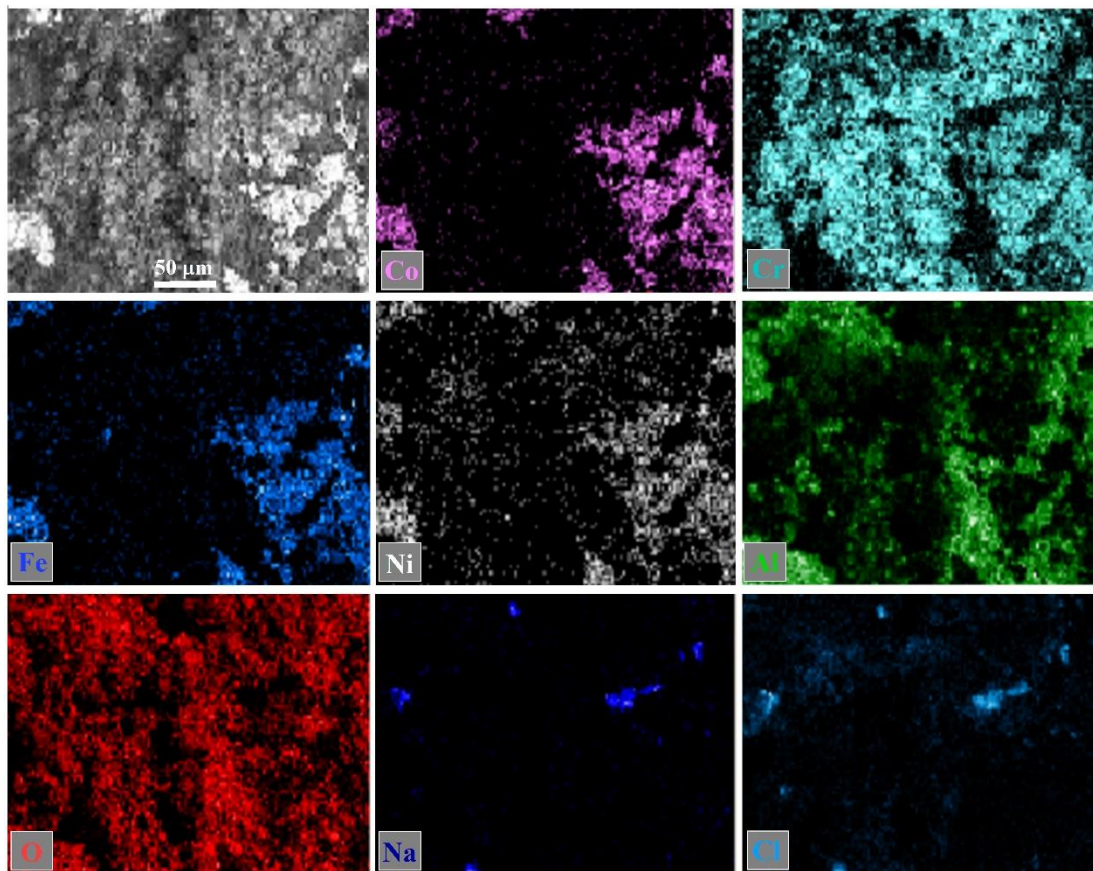


Fig. 4. Elemental mapping analysis of the hot corroded CoCrFeNiAl HEA in NaCl molten salt at 850°C for 120 h

The cross-sectional microstructure of corrosion scale that formed following the corrosion test in NaCl molten salt at 850°C for 120 h is shown in Fig. 5 (a). Fig. 5 (b and c) corresponds to EDS point analysis and elemental mapping of the cross-sectional micrograph. By considering the cross-sectional micrograph of the scale in combination with the corresponding elemental distribution maps, it follows that the corrosion scale formed on the alloy consists of layers, which are enriched with different elements. Based on the EDS result for point 1, the top layer is mainly enriched with Cr and O, indicating the constitution of Cr₂O₃ on the scale. Beneath the top layer (point 2), the content of Cr in the scale decreases, whereas that of Al increases. Besides, Na and Cl were found along with oxygen in this layer. Point 3 which lies on the inner layer consists largely of O and Al

corrosion scale formed on the alloy appeared to have a porous and loose feature, most probably due to the NaCl molten salt.

When the hot corrosion mechanism is evaluated in general, the hot corrosion mechanism is significantly different from that of simple air oxidation. Mohanty et al. [21] have stated that the material degradation is increased about 200 times in the presence of sulfate contaminants as compared to oxidation in air, however, it is increased about 2000 times by mixtures of sulfate and chloride contaminants. It is important to emphasize that NaCl is the only corrosive agent used in this work, thus it is expected to cause the intensive degradation of the alloy under the adopted experimental conditions. Furthermore,

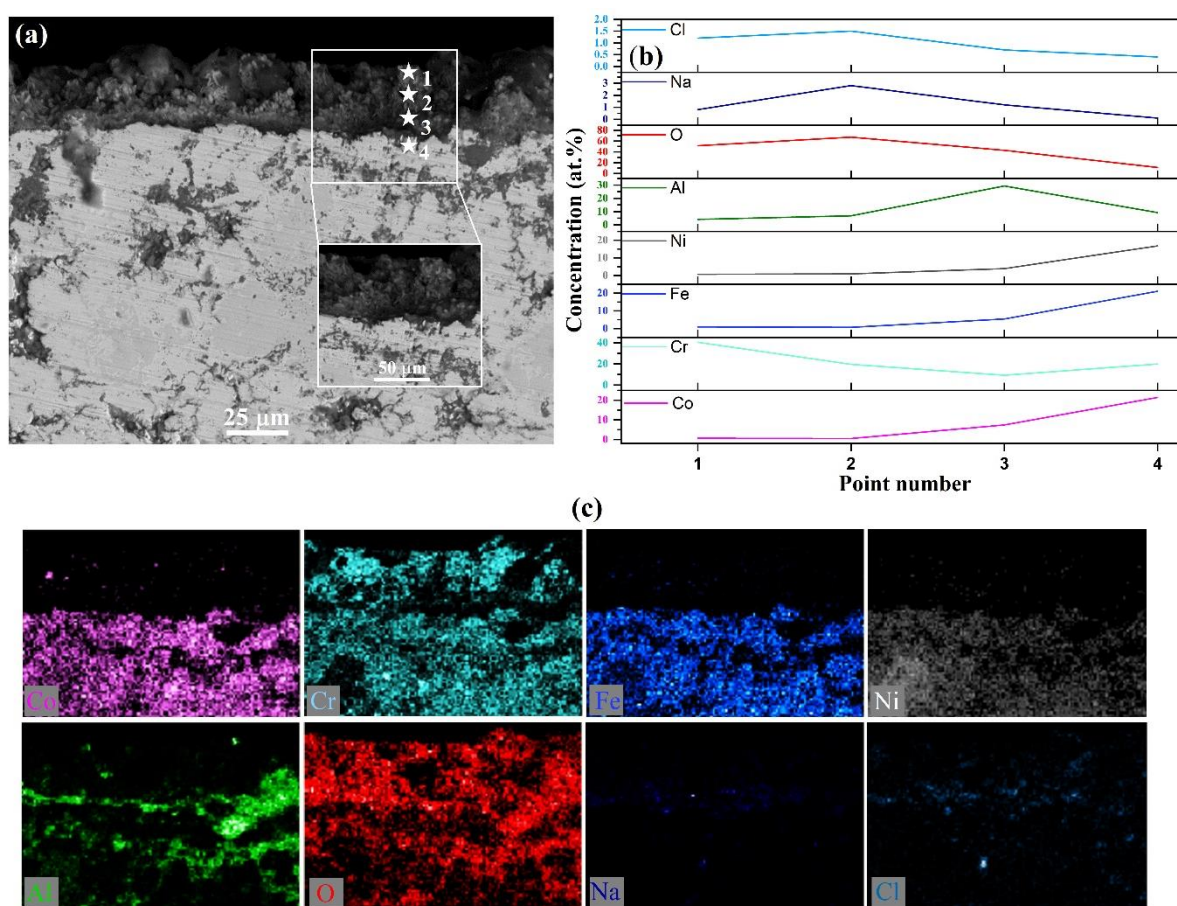
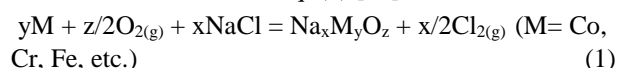
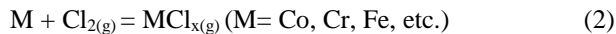


Fig. 5. Cross-sectional micrograph (a) EDS analyses along points 1-4 (b) and elemental mapping (c) of the hot corroded CoCrFeNiAl HEA in NaCl molten salt at 850°C for 120 h

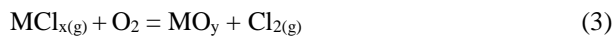
and a moderate enrichment of Co, Cr, Fe and Ni can be noticed. This layer corresponds to the formation of Al₂O₃, as proved by elemental distribution maps and XRD pattern. Oxygen response is witnessed within the substrate, suggesting that the scale does not effectively impede the oxygen ingress. It is important to point out that the Cr₂O₃ layer constitutes a significant portion of the corrosion scale formed on the surface. After corrosion at 850°C for 120 h, the total scale thickness formed on the alloy was found to be around 32 µm. Moreover, the

the corrosion mechanism becomes more comprehensive in the presence of molten salt because many different electrochemical reactions can appear. This is caused owing to the fact that it involves consideration of chemical reactions between the molten salt and corrosion products. It is generally thought that the hot corrosion in the presence of NaCl salt is initiated by oxychlorination reaction, as described in Eq. (1) [22]:





At the same time, chlorine released from the above reaction (1) plays an important role in provoking the chlorination reaction (2). This reaction indicates that the chlorine diffuses inward through the microcracks, voids and grain boundaries, and reacts with the constituent elements of CoCrFeNiAl HEA to form the volatile chlorides. Then, these volatile chlorides (such as $AlCl_x$, $CrCl_x$, $FeCl_x$) will volatilize through the same route to the outer surface, where the oxygen activity is higher. Here, the volatile chlorides will oxidize because their corresponding oxides are thermodynamically more stable according to the following reaction [16]:



Some of the chloride gas re-generated via reaction (3) will penetrate into the substrate, afterward attack the alloy components to form volatile MCl_x species again, as exemplified by Eq. (2). Consequently, as the hot corrosion progresses, the degradation extent of the studied HEA becomes more critical because the chlorination/oxidation reactions ((2) and (3)) occur cyclically, leading to a self-sustaining process. That is to say, the released chlorine gases played a major role in driving the hot corrosion behavior of the studied HEA. The detection of Cl throughout the corrosion scale proves that self-sustained cyclic reactions occur, as evidenced in elemental mapping shown in Fig. 5 (c). It is possible that the cyclic chlorination/oxidation reactions may be responsible for the formation of pores and corrosion voids visible in Fig. 5 (a). Further to say, the presence of chlorine not only causes these defects in the scale but also brings about poor adherence of the corrosion products associated with mass loss, which results in a severe reduction in corrosion resistance of the alloy. Therefore, it can be concluded that the compactness and continuity of the scale are damaged due to the sodium chloride-induced hot corrosion.

4. CONCLUSION

The present work dealt with the hot corrosion behavior of CoCrFeNiAl HEA produced by powder metallurgy in NaCl molten salt at 850°C for 120 h. The main conclusions based on the obtained experimental results can be drawn as follows:

1. After 120 h of hot corrosion at 850°C, the total mass gain of CoCrFeNiAl HEA was about 2.2 mg/cm².
2. It has been shown that the corrosion products formed on the HEA are composed of Al_2O_3 , Fe_2O_3 , Cr_2O_3 , NiO and $NiCr_2O_4$.
3. A corrosion scale with a mean thickness of 32 μm after 120 h of hot corrosion was found on the HEA surface.
4. The corrosion scale with a multi-layered structure featured poor adhesion to the substrate. The scale showed porous and loose attributes making it more susceptible to further hot corrosion.

DECLARATION OF ETHICAL STANDARDS

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Yiğit GARİP: Performed the experiment studies, analyzed the results and wrote the manuscript.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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