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RESEARCH ARTICLE

Influence of equal channel angular pressing on the strength and corrosion properties of FeNiMnCr high-entropy alloy

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Abstract

Objectives. High-entropy alloys (HEAs) represent a novel class of metallic materials known for their exceptional mechanical and corrosion-resistant properties. This study investigates the effects of equal channel angular pressing (ECAP) on the microstructure, tensile strength, and corrosion behavior of an equiatomic FeNiMnCr alloy.

Methods. The alloy was synthesized via arc melting, homogenized, and subjected to up to four ECAP passes at 400°C. Phase composition was analyzed using X-ray diffraction, while microstructural features were examined using scanning electron microscopy and transmission electron microscopy. Mechanical properties were evaluated based on Vickers microhardness and tensile testing, while corrosion resistance was assessed in a 3.5% NaCl solution using potentiodynamic polarization.

Results. The results indicate a significant grain refinement, an increased hardness and strength (by 1013 MPa), and an improved corrosion resistance of the alloy after ECAP processing.

Conclusions. The study demonstrates that ECAP is an effective method for enhancing the performance of FeNiMnCr HEAs. This makes it promising for use in nuclear energy, medicine, and aerospace industry.

Keywords

high-entropy alloy, equal channel angular pressing, strength, thermal stability, corrosion resistance

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НАУЧНАЯ СТАТЬЯ

Влияние равноканального углового прессования на прочностные и коррозионные свойства высокоэнтропийного сплава FeNiMnCr

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Аннотация

Цели. Исследовать влияние равноканального углового прессования (РКУП) на структуру, прочность и коррозионные свойства сплава FeNiMnCr.

Методы. Структурные характеристики изучались с помощью рентгенофазового анализа и электронной микроскопии (scanning electron microscopy, transmission electron microscopy). Механические свойства оценивались по микротвердости и испытаниям на растяжение, коррозионная стойкость — потенциодинамическим методом в 3.5% растворе NaCl.

Результаты. Установлено, что РКУП приводит к значительному измельчению зерна, увеличению прочности (до 1010 МПа) и снижению плотности коррозионного тока, что говорит об улучшении пассивирующих свойств поверхности.

Выводы. РКУП повышает прочность и коррозионную стойкость сплава, что делает его перспективным для применения в ядерной энергетике, медицине и авиационно-космической промышленности.

Ключевые слова

высокоэнтропийный сплав, равноканальное угловое прессование, прочность, термическая стабильность, коррозионная стойкость

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INTRODUCTION

High-entropy alloys (HEAs) constitute a novel class of metallic materials characterized by the presence of five or more principal elements in near-equiatomic proportions [1–4]. This unique compositional strategy often results in exceptional mechanical strength and corrosion resistance, making HEAs a subject of extensive global research. Despite their promising properties, many HEAs suffer from limited strength, which restricts their practical applications. To overcome this limitation, various processing techniques can be employed to enhance the mechanical properties of such alloys.

The conventional approach to creating new structural materials involves selecting one main element as a matrix and its further alloying to obtain the desired combination of mechanical and/or technological properties. Recently, a new approach to creating multicomponent alloys containing several elements in nearly equiatomic concentrations has been proposed. In comparison with traditional alloys, these alloys are characterized by higher mixing entropy values, hence the name HEAs. The increased entropy in HEAs is explained by the maximum mixing entropy between dissolved components upon their equiatomic concentration. As a result, single-phase solid solutions with simple body-centered cubic (BCC) or face-centered cubic (FCC) lattices are formed in several HEAs. Depending on the chemical composition, HEAs can demonstrate enhanced functional properties, such as hardness, wear resistance, thermal stability, corrosion resistance, and superplasticity [5–10]. Meanwhile, methods of severe plastic deformation (SPD) offer additional possibilities for regulating the functional properties of metals and alloys by forming an ultrafine-grained structure. SPD methods are known to be capable of reducing the grain size to less than 300 nm in various metals and alloys [11], while the use of heat treatment (HT) on ultrafine-grained samples can further increase their strength and endurance limit. The high mixing entropy of elements in the alloy is considered a measure of the probability of maintaining their system in this state. This ensures an increased thermal stability of the phase composition and structural state, along with the mechanical, physical, and chemical properties of the alloy. Thus, HEAs demonstrate the potential for forming and maintaining a multielement solid solution both immediately after crystallization and during subsequent thermomechanical treatment, acquiring unique combinations of characteristics [5–10].

A number of reviews [1–4, 12] present the research results on the structure and properties of HEAs; however, they mainly provide experimental data for coarse-grained and cast samples. One work reported the use of SPD for AlCrFeCoNiCu HEAs, but considered only structural-phase transformations during deformation processing.

The emerging interest in the application of SPD methods has not bypassed HEA materials, promising to enhance their functional properties. However, many HEAs contain Co in significant concentrations, which is undesirable for their use in nuclear reactors from the perspective of neutron activation and radiation waste management. Therefore, a new single-phase FeMnNiCr alloy with Co replaced by Mn, showing significant radiation resistance, has recently appeared [13]. Among HEAs, the FeNiMnCr system is known to form a stable FCC structure and to exhibit a good balance of strength, ductility, and corrosion resistance. However, further enhancement of its properties is essential for advanced engineering applications. Equal channel angular pressing (ECAP), a severe plastic deformation technique, offers a promising approach to improving the mechanical and corrosion characteristics of alloys without altering their chemical composition.

In this study, we aim to evaluate the influence of ECAP processing on the structural, mechanical, and corrosion properties of the FeNiMnCr alloy.

EXPERIMENTAL

The investigated HEA had a nominal composition of $Fe_{30}Ni_{30}Mn_{30}Cr_{10}$, which was selected due to its single-phase FCC structure as well as promising mechanical and corrosion properties. The alloy was synthesized by arc melting of high-purity elemental metals (purity ≥ 99.9%, University of Missouri, USA) in an argon atmosphere using a nonconsumable tungsten electrode (USA). To ensure chemical homogeneity, the ingot was remelted at least five times and flipped between each melting cycle. The final as-cast ingots were subjected to homogenization at 1100°C for 12 h in an evacuated quartz tube, followed by water quenching.

The billets (cylinders 20 mm in diameter and 100 mm in length) were processed via ECAP using a die with an internal channel angle of 120°, following the Bc route. Processing was conducted at 450°C for up to three passes to refine the microstructure and enhance strength. The deformation speed was approximately 1 mm/s.

The chemical composition of the alloy was confirmed by energy-dispersive X-ray spectroscopy (EDS). The phase composition was analyzed using X-ray diffraction (XRD) using a Bruker D2 Phaser diffractometer (*Bruker AXS GmbH*, Germany) with CuK_α radiation (0.154 nm) over a 2θ range of 20°–100°, both prior to and following ECAP. The analysis of XRD patterns was carried out using the Rietveld method implemented in the MAUD software package (University of Trento, Italy). The microstructure was studied using a TESCAN MIRA scanning electron microscope (SEM) (*TESCAN ORSAY HOLDING*, Czech Republic) and a JEM-2100 transmission electron microscope (*JEOL*, Japan) with an accelerating voltage of 200 kV, equipped with an attachment for EDS analysis, with special attention to grain size and phase distribution.

Mechanical properties were evaluated via microhardness testing by a Micromet 5101 device (*Buehler*, USA) (100 g load, 10 s dwell time) and tensile tests at room temperature (the range of 20–25°C (293–298 K)) with a strain rate of 10^{-3} (testing machine Instron 8801 — *Instron*, USA / United Kingdom). Corrosion resistance was assessed using potentiodynamic polarization in 3.5 wt % NaCl solution at 37°C using an R-5X electrochemical station (*Elins*, Russia). Electrode potential measurements were conducted for 2 h to achieve a steady-state value. Potentiodynamic polarization (PDP) was performed across the range from −600 to +400 mV relative to the open circuit potential at a scanning rate of 0.25 mV/s. A silver/silver chloride electrode filled with a 3.5 M KCl solution was used as a reference. The counter electrode was a graphite rod. PDP results were calculated using the Tafel method. Polarization resistance R_p was calculated from the slope of the polarization curve ±10 mV relative to the free corrosion potential.

RESULTS AND DISCUSSION

Figure 1a shows the alloy structure in the initial state, which is characterized by large grains with an average size of 290 µm. According to EDS analysis, the chemical composition of the alloy contains Fe = 30.93 wt %, Ni = 31.18 wt %, Mn = 29.57 wt %, and Cr = 8.32 wt %. In addition, segregations of Ni and Mn atoms near the grain boundaries are observed (Fig. 1). At the same time, there are also particles of NiMn precipitates in the grains, mainly of a globular shape with an average size of ~23 µm (Fig. 1b). EDS analysis showed that the content of Cr atoms in both triple junctions and particles was significantly lower, while the content of Mn and Ni was significantly higher, in comparison with the equilibrium content of these elements in the studied alloy Fe 30 wt %–Ni 30 wt %–Mn 30 wt %–Cr 10 wt % (points 1 and 2, Figs. 1b).

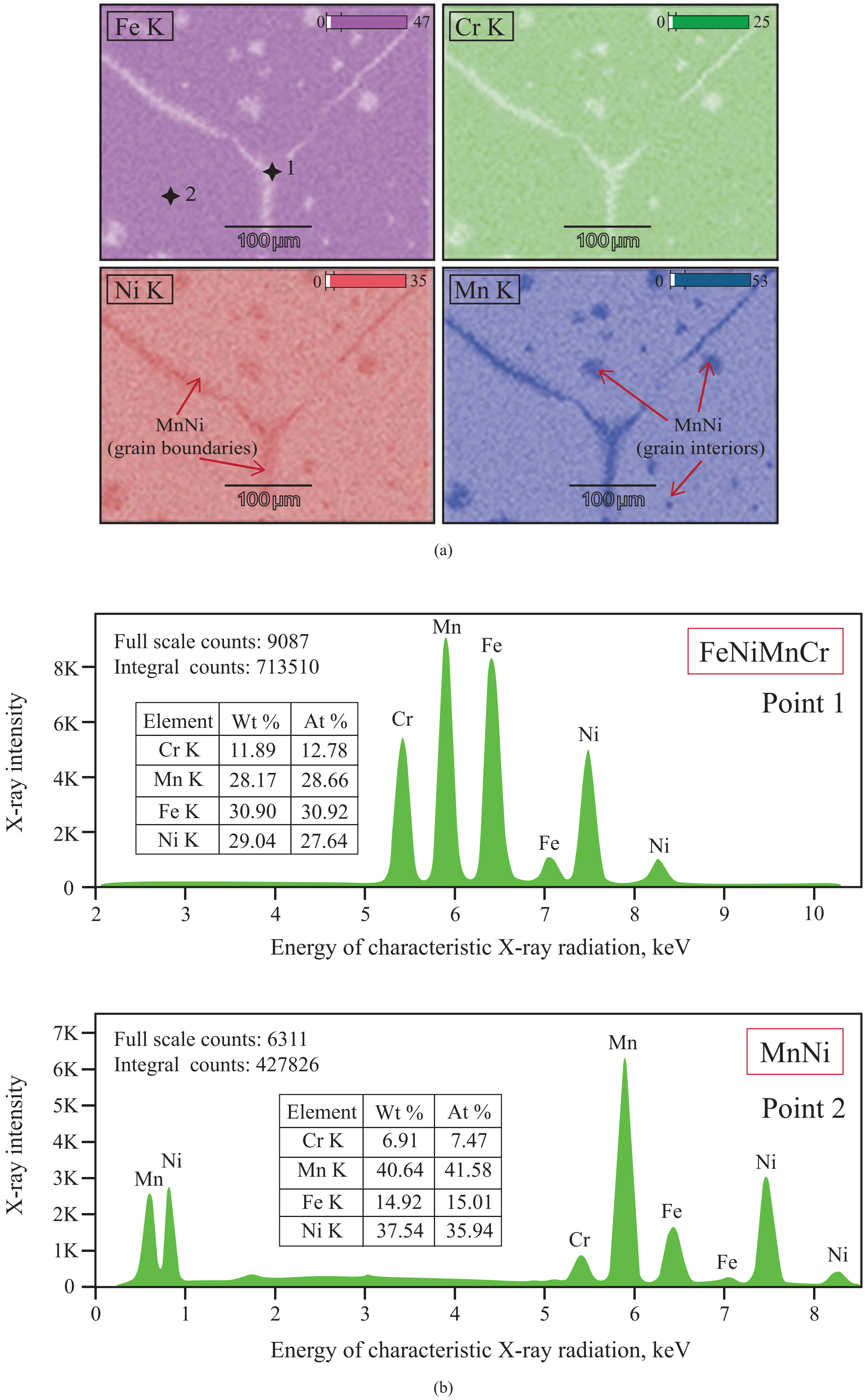

Fig. 1. EDS image of the initial alloy microstructure: (a) elemental distribution in the initial state; (b) elemental analysis at the triple junction (point 1) and in the MnNi particle (point 2)

Thermal stability studies showed the ECAP sample to exhibit the highest microhardness of 3500 MPa (Fig. 2), which was 80% higher than that of the sample in its initial state. The high thermal stability up to a temperature of 600°C is apparently due to conducting the ECAP deformation processing of the alloy at an elevated temperature of 450°C followed by annealing at 550°C (ECAP450°C+HT550°C).

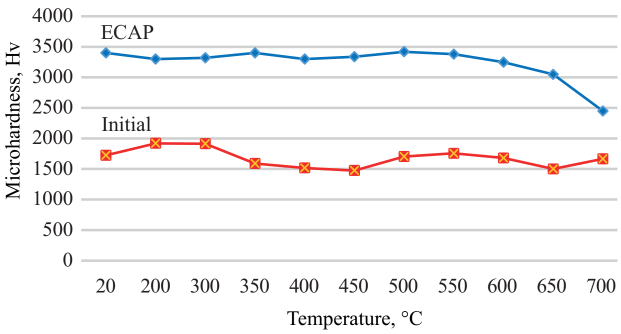

Fig. 2. Microhardness dependence on annealing temperature

In the sample subjected to ECAP+HT550°C processing, the transmission electron microscopy images revealed the presence of separate regions with elongated grains with a width of 0.3–0.5 μm and a length of 0.5–1.5 μm, within which an increased dislocation density was observed (Fig. 3).

X-ray diffraction (XRD) patterns of the FeNiMnCr alloy in the as-cast and ECAP-processed states are shown in Fig. 4. Their analysis showed that, both in the initial state and after the ECAP450°C+HT550°C treatment, the sample contains the FCC phase FeNiMnCr and the secondary phase MnNi (Fig. 4). The reflections of these phases are highlighted in Fig. 4 by blue rhombs and red squares at the bottom of the XRD patterns. After ECAP processing, the FCC phase remained dominant; however, the peaks became broader and slightly shifted. Peak broadening is associated with a significant grain refinement and the accumulation of internal lattice strain due to severe plastic deformation. No new phases or intermetallic compounds were

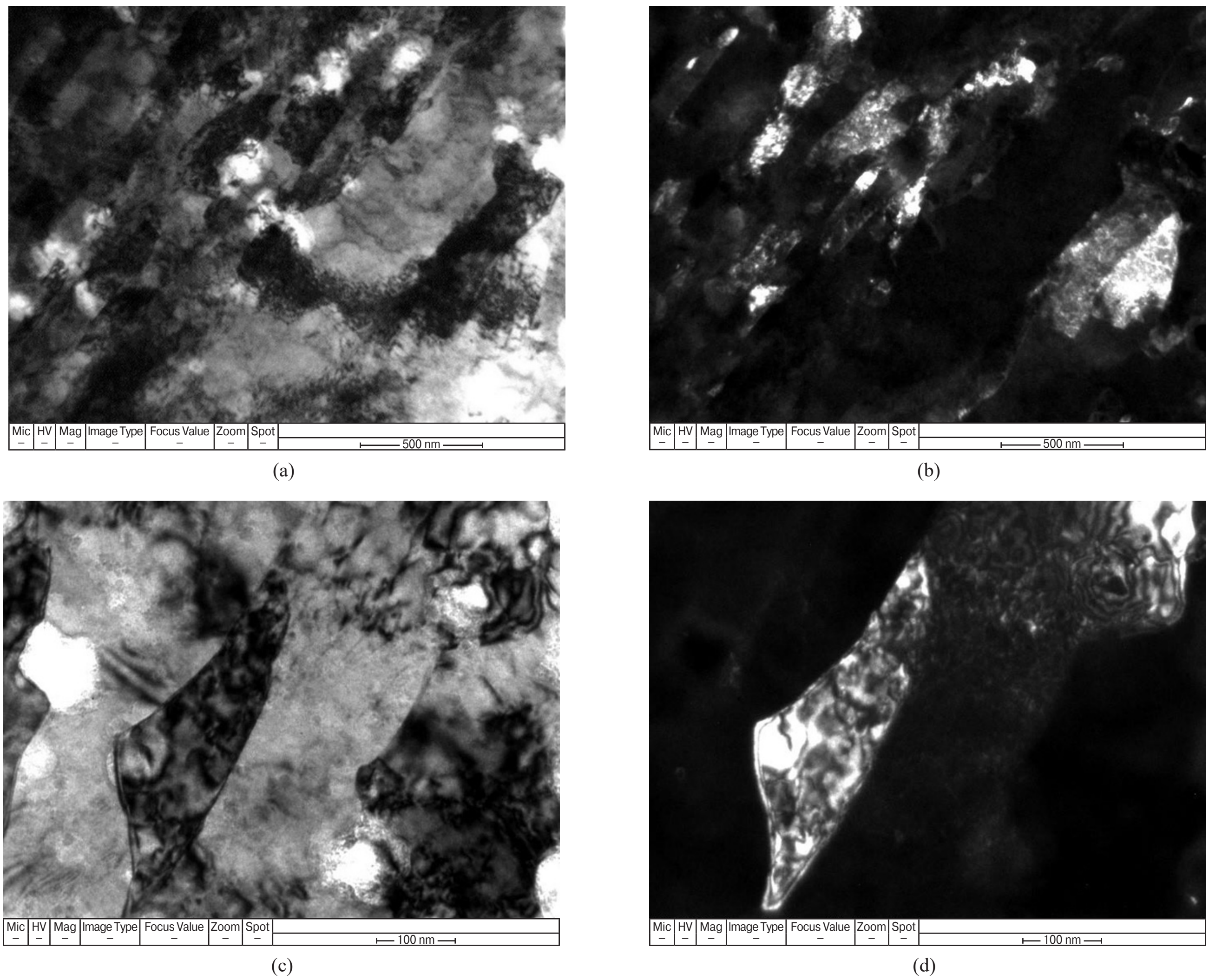

Fig. 3. Various regions in the alloy structure after ECAP+HT550°C, observed by a transmission electron microscope: (a, c) bright-field image; (b, d) dark-field image

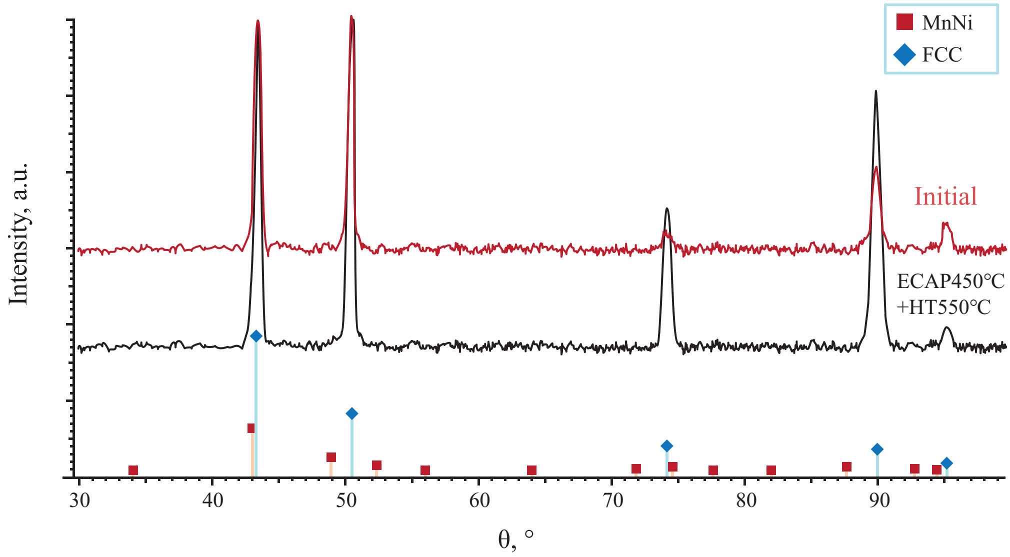

Fig. 4. X-ray diffraction patterns

detected, confirming the structural stability of the alloy under ECAP conditions.

The lattice parameter of the as-cast alloy (3.6158 Å) was calculated using the Bragg equation and was found to increase slightly after ECAP (3.6211 Å), which may be attributed to the redistribution of interatomic distances along numerous grain boundaries due to local strains.

The experimental data from tensile tests were used to construct graphs showing the dependence of strain on stress, as presented in Fig. 5. Prior to testing, the initial and ECAP samples were additionally annealed at temperatures of 450°C and 550°C, respectively, to relieve internal stresses. The tensile tests of the ECAP+550°C sample established the ultimate tensile strength to be 1013 MPa, which increased by more than 2.5-fold compared to that in the initial sample (377 MPa) annealed at 450°C. Meanwhile, the elongation of the initial sample was 63%, whereas it was much lower for the ECAP sample, at the level of 3.6%.

Figure 6 shows the results of electrochemical tests in the form of polarization curves.

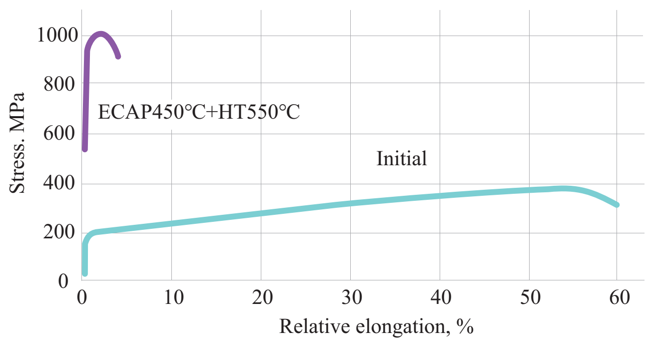

Fig. 5. Results of tensile tests

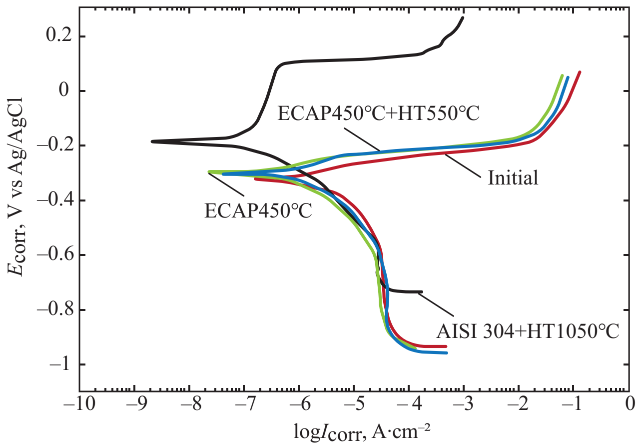

Fig. 6. Polarization curves obtained from electrochemical tests. E_{corr} is an open-circuit potential; I_{corr} is the lowest corrosion current

The table presents the corrosion parameters of the samples calculated based on electrochemical tests and corresponding polarization curves.

Table. Corrosion parameters

Sample	E_{corr}, V	I_{corr}, A/cm^2	R_p, Ohm·cm^2
Initial+450°C	−0.361 ± ± 0.12	$1.91 \cdot 10^{-6}$ ± ± $1.12 \cdot 10^{-7}$	$2.15 \cdot 10^4$ ± ± $0.23 \cdot 10^4$
ECAP450°C	−0.300 ± ± 0.01	$1.21 \cdot 10^{-6}$ ± ± $1.28 \cdot 10^{-7}$	$3.44 \cdot 10^4$ ± ± $0.41 \cdot 10^4$
ECAP450°C+HP550°C	−0.306 ± ± 0.01	$1.28 \cdot 10^{-6}$ ± ± $1.25 \cdot 10^{-7}$	$2.13 \cdot 10^4$ ± ± $0.11 \cdot 10^4$
AISI 304L	−0.190 ± ± 0.01	$0.15 \cdot 10^{-6}$ ± ± $1.24 \cdot 10^{-7}$	$23.3 \cdot 10^4$ ± ± $0.11 \cdot 10^4$

It can be seen from the table that processing conditions have a significant effect on the open-circuit potential (E_{corr}). A higher E_{corr} value indicates surface passivation, while a lower value indicates surface activation. Among the HEA samples, the ECAP sample showed the highest corrosion resistance (the lowest corrosion current I_{corr}). After additional annealing at 550°C (Table), the corrosion current slightly increased, although remaining lower than that in the initial state. The decrease in corrosion current and the increase in polarization resistance (R_p) after ECAP processing compared to the initial sample indicate better surface passivation in the ECAP samples during testing. Additional HT of the ECAP samples at 550°C led to the preservation of the corrosion current and open-circuit potential at the corrosion resistance level of the ECAP sample, and the maintenance of polarization resistance at the level of the initial samples. It can be assumed that the passive film formed on the surface of the ECAP HEA samples exhibits a greater adhesion to the crystal lattice defects in the form of grain boundaries with respect to the initial state. For comparison, Table also shows the corrosion parameters of AISI 304L stainless steel tested under identical conditions. This steel demonstrated a significantly lower corrosion current and a higher polarization resistance compared to the HEA samples, indicating a more passivated surface than the other samples. AISI 304L stainless steel was tested also under 3.5 wt % NaCl solution with potentiodynamic polarization in [14]. Overall, it should be noted that in the studied HEA, grain refinement by ECAP increases the ultimate tensile strength by more than 2.5 times, while maintaining corrosion resistance at the level of the initial samples.

In order to further understand the enhancing effect of ECAP on the mechanical and corrosion properties of the FeNiMnCr HEA, an additional analysis was conducted.

The increase in ultimate tensile strength and microhardness observed in ECAP+550°C samples is primarily attributed to the significant grain refinement resulting from severe plastic deformation. Transmission electron microscopy confirmed the presence of ultrafine elongated grains and high dislocation density, both of which contribute to strengthening via grain boundary strengthening and dislocation interaction mechanisms. The increased dislocation density enhances strain hardening and impedes dislocation motion, thus leading to elevated strength.

The corrosion behavior is also positively influenced by the ECAP process. The refinement of grain structure results in a higher density of grain boundaries, which are known to serve as preferential sites for passive film nucleation. This likely improves the adhesion and stability of the protective oxide layer, thereby reducing the corrosion current density and increasing polarization resistance. This is consistent with the results reported in recent studies [15, 16], which also observed improvements in corrosion resistance due to fine microstructural features.

Our findings align with [17], which emphasized the importance of phase composition and deformation-induced effects in enhancing the mechanical performance of HEAs. However, our study uniquely demonstrates these improvements in a cobalt-free FeNiMnCr system, which is particularly relevant for radiation-sensitive environments.

Improved characteristics of ECAP samples of HEA are of interest for the development of technologies for producing materials with optimal performance characteristics for operation under elevated temperatures and aggressive environments in nuclear power engineering, in the aerospace industry, for the manufacture of parts with high strength and thermal stability, as well as in medicine, due to good corrosion resistance and biocompatibility.

CONCLUSIONS

The $Fe_{30}Ni_{30}Mn_{30}Cr_{10}$ HEA demonstrates remarkable enhancements in both mechanical and corrosion properties when subjected to ECAP. The experimental results revealed that the ultimate tensile strength of the alloy increased from 377 MPa in its initial state to 1013 MPa after ECAP and subsequent HT, while its microhardness improved by 80%, reaching 3500 MPa. Additionally, the corrosion resistance of the alloy remained robust, with the ECAP samples exhibiting a lower corrosion current and higher polarization resistance compared to their initial state, indicating improved surface passivation.

These improvements are primarily attributed to the formation of an ultrafine-grained structure and increased dislocation density, which enhance strength through grain boundary and dislocation strengthening mechanisms. The refined grain structure also promotes the formation of a more stable and adherent passive film, contributing to improved corrosion resistance.

Furthermore, the cobalt-free composition of the alloy and its demonstrated stability under thermal and corrosive conditions render it a promising candidate for applications in the nuclear industry, chemical processing, and marine environments where high strength and corrosion resistance are critical under extreme conditions.

These results confirm that precise thermomechanical treatments of HEAs, such as FeNiMnCr, can enhance their performance for critical applications in industries where materials are exposed to extreme conditions. Our findings emphasize the potential of tailored processing techniques in improving HEA properties for advanced engineering applications.

Authors' contribution

A.M.M. Abuayash — investigation of mechanical properties.

K.M. Nesterov — research of corrosion resistance.

R.K. Islamgaliev — structural investigations.

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