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Single-phase equiatomic Co-Cr-Fe-Ni high-entropy alloy with excellent strain gauge sensitivity

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Abstract

Single-phase Co-Cr-Fe-Ni high-entropy alloys boast exceptional mechanical and electrical properties. These alloys are suitable for various functional applications, particularly as strain gauges and high ohmic resistors, due to this fact. The resistance-tension tests of the equiatomic CoCrFeNi high-entropy alloy demonstrate higher strain gauge factor (GF) and elastic limit values than commercial strain gauges. In the elongation range of ϵ = 0–0.2%, the resistivity of the studied alloys follows a strictly linear pattern with respect to the applied load, up to 435 MPa. Heat treatment enhances the GF value of the as-cast alloy from 3.07 to 3.45. The annealing process changes the sample structure, resulting in an increase in the GF.

Keywords

CoCrFeNi high-entropy alloy strain gauge structure electrical resistivity

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1. Introduction

High-entropy alloys, unlike traditional alloys and steels, are multicomponent systems [1, 2] without a dominant element. 20 years after their discovery, HEAs continue to garner significant interest due to their versatility as both functional and structural materials. Recent studies have shown [3] that such applications may include strain gauges and high ohmic resistors based on refractory or 3d metals. The tensile strength values of 1100-1500 MPa and plasticity reaching a value of 63-71% at room temperature, weak linear changes in electrical resistivity depending on temperature and tension [4, 5], and the paramagnetic state [6] above room temperature allows us to consider HEAs as a replacement for commercial strain gauges. The unique structure of HEAs enables the favorable combination of above-mentioned properties. In such systems, BCC, FCC, or FCC-structured single-phase solid solutions are stabilized by augmented configurational entropy [1]. In addition, in high-entropy alloys, crystal lattices exhibit disordered and defective structures because of differences in the atomic radii and valences. Configurational entropy and chemical complexity are essential features of equiatomic high-entropy alloys.

Here, we selected equiatomic CoCrFeNi alloy for research, guided by the following reasons: 1) it forms a single-phase solid solution with a simple FCC crystal structure [7]; 2) it is paramagnetic [8] above 100 K, so there is no influence of the magnetic component on the electron and lattice subsystem; 3) it has high electrical resistivity, low temperature resistivity and thermal expansion coefficients [5, 9]. Thus, the equiatomic CoCrFeNi alloy is an ideal system for studying the influence of heat treatment and structure on strain gauge and electrical resistivity.

In our study, we selected an equiatomic CoCrFeNi alloy and for the first time determined its strain sensitivity as one of the most important parameters indicating the prospects for further consideration of this alloy as a pressure and mechanical deformation sensor. The purpose of this work was to determine the functional applicability of the equiatomic CoCrFeNi alloy to baric and/or strain gauge applications.

2. Experimental procedure

Initially, the as-cast ingot was produced by arc melting on a water-cooled furnace mold from appropriate quantities of Ni, Co, Fe and Cr (>99.9% purity). The ingot was turned over and remelted five times in high-purity argon and then

subjected to thermal treatment as described below. The sample was annealed in a resistance furnace at 900 °C for 24 h under an inert environment. The heating speed was 3K/min, the cooling speed – 10 K/min. The YUMO B-9M device was used to cold-roll the foils. The ingots were cold-rolled to a thickness of 200 μm , the deformation of the sample was approximately 98%. 200×4×0.2 mm foils, both ascast and annealed, underwent resistance-tension tests.

Resistivity testing was performed on an original automated device following the application of a load. The electrical resistivity data were recorded using a standard 4-contact method with a DC current source AKIP-2101 and AKIP-6304 resistivity meter. The device was calibrated using standard alloys manganin, constantan, and nichrome for strain gauges. Three sets of measurements were performed on each sample until the foils were ruined.

To estimate the GF, we use the following relation:

$$GF = \frac{\Delta R/R_0}{\varepsilon} \tag{1}$$

where $\Delta R/R_0$ is the relative resistivity and ε is the relative elongation, %.

The relative elongation was calculated using Hooke's law as follows:

$$\varepsilon = \frac{\sigma}{F'} \tag{2}$$

where σ is the cross-sectional strain measured by the direct method, MPa, and E is the Young's modulus, MPa, whose value for equiatomic CoCrFeNi alloy was obtained from experimental data [9].

A Shimadzu XRD-7000 diffractometer, a LOMO MMU-3 microscope with a SONY ICX452AQ CCD camera, and a Carl Zeiss EVO 40 electron microscope fitted with an EDS INCA X-Act Energy microanalyzer were employed to examine the crystal structure, microstructure, and local chemical composition of the sample surfaces.

3. Result and Discussion

The X-ray diffraction patterns, images and unit cell parameters for the as-cast (A) and annealed (B) pre- and post-cold rolling alloys are presented in Figure 1 and Table 1. A and B samples only show FCC symmetry peaks. Based on the XRD analysis, all samples are identified as single-phase FCC solid solutions. The unit cell parameters remain practically unchanged. The SEM-EDS mapping results (Figure 2) indicate that the alloy compositions closely match their nominal values. Along the (200) plane, A sample exhibit a texture with a preferential orientation. Rolled A and B samples demonstrate texturing along (111) and (220) plane. The images are consistent with their corresponding XRD patterns. As shown in Figure 1, the as-cast sample features both large and small elongated grains, 100-300 µm wide large grains are needle-shaped with lengths of 800-1200 μm , while the small grains are 100-300 μm in size. The elongated grains, texture is oriented toward the heat flow during solidification. At higher magnification, coarse grains appear as assemblages of columnar blocks of diverse sizes and orientations. Annealing increases in size and a shift toward an oval shape for these columnar blocks (Figure 1). The grain size of \boldsymbol{B} sample is about 1000–1200 μm , and the grain shape is close to equiaxed. The crystallite sizes are 2–4 μm , so the structure of the annealed sample can be classified as fine-grained. The microstructure of the foil from sample \boldsymbol{A} is represented by crystallites of non-uniform shape and size. The crystallites in \boldsymbol{B} sample exhibit a fine equal-band structure of continuous length. Twinning, which occurs under cold rolling conditions, promotes reorientation of columns along the rolling axis. In this case, grains with (111) and (220) plane prevail on the surface sample.

The dependence of relative resistivity $\Delta R/R_0$ versus ϵ for the cold rolled **A** and **B** foil and the GF values are displayed in Figure 3.

Table 1 Unit cell parameters of cubic phases the CoCrFeNi alloys.

Alloy	As-cast	As-cast cold-rolled	Annealed	Annelead cold-rolled
Unit cell a, Å	3.580(1)	3.574(1)	3.575(1)	3.576(1)

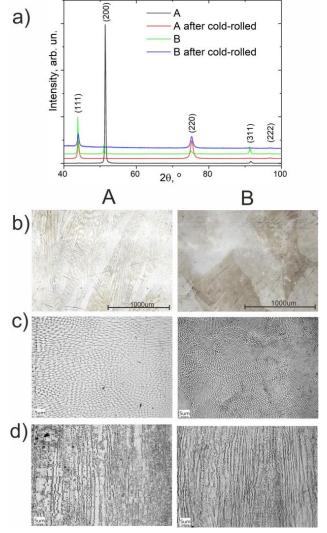


Figure 1 XRD patterns (a) and images (b, c, d) of the equiatomic CoCrFeNi alloy in the as-cast (A), annealed (B) and cold rolled (d) condition.



Figure 2 Elemental maps of Cr, Fe, Co and Ni for the as-cast ${\tt CoCrFeNi}$ alloy.

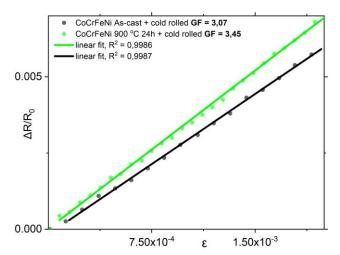


Figure 3 Relative resistivity $\Delta R/R_o$ versus strain ϵ for the cold-rolled CoCrFeNi alloy in as-cast (A) and annealed (B) states.

The resistivity shows a linear response to an applied load up to ϵ = 0.2%, while above this point the response becomes nonlinear because the elastic limit σ_y is reached. For the **A** and **B** foil, the specific electrical resistance ρ , gauge factor and elastic limit σ_y at room temperature are collected in Table 2.

According to the data from Table 1, GF rose by 12% from 3.01 to 3.45. The GF increase is attributable to the evolution of the microstructure due to thermal treatment. Heat treatment at 900 $^{\circ}$ C enlarges grain sizes after cooling. The microstructure becomes uniformly fine-grained at the same time.

Widely used commercial strain gauges such as those made of manganin (GF = 0.5-0.7) [10], constantan (GF = 1-3) [10, 11] and nichrome (GF = 1.2-2.7) [12,13] were discovered more than a hundred years ago. Despite their lower GF and σ_y , compared to modern strain gauges based on platinum, gold and palladium [14, 15], they offer a significant cost advantage. The 3d classical metal alloy strain gauges come with a low elastic limit of 300–350 MPa and a limited ϵ range where $\Delta R/R_0(\epsilon)$ dependence is linear. They are unsuitable for use under loads exceeding 400 MPa. Commercial strain gauge alloys are metallic systems based on one or two elements that cannot compete with HEAs for many reasons.

Table 2 Specific electrical resistivity $\rho,$ gauge factor (GF) and elastic limit $\sigma_y.$

ρ (μΩ·cm)	GF	σ_y (MPa)
79±2	3.07±0.30	450±33
76±2	3.45±0.33	435±15
43-44	0.47-0.71	300-400
100-200	1.20-2.70	-
45-50	1.0-3.0	302
59	3.7-4.0	-
112	5.1-6.6	513
	79±2 76±2 43-44 100-200 45-50 59	79±2 3.07±0.30 76±2 3.45±0.33 43-44 0.47-0.71 100-200 1.20-2.70 45-50 1.0-3.0 59 3.7-4.0

The potential strain gauge applications of HEAs are due to several favorable circumstances. Many HEAs form single-phase, thermally stable solid solutions. These systems are characterized by a unique combination of ductility and high corrosion resistance, which is extremely important for strain gauge applications. An increase in the strain sensitivity coefficient after cold rolling compared with classical strain gauges occurs due to the crystallographic anisotropy of the FCC lattice and the texture. The presence of texture in the as-cast sample positively affected the tensile strength, creep, corrosion resistance, and fatigue characteristics of the cold-rolled high-entropy alloy. These structural features ultimately significantly increase the HEAs strain sensitivity, which is greater than that of commercial binary alloys. For example, in the binary alloy NiCr (nichrome), ordering effects may occur, which leads to the formation of a phase with a Ni₂Cr supercell or a cluster structure based on Cr [16]. The formation of secondary phases or ordered cluster structures negatively affects the strain sensitivity due to an increase in the brittleness of the Ni-Cr alloy.

The success of employing materials as strain gauges hinges on their sensitivity gauge factor. The electrical resistivity must remain strictly linear with respect to both the applied load and temperature. The cold-rolled equiatomic CoCrFeNi alloy exhibited a strain sensitivity coefficient of 3.45 and a linearly increasing resistivity under loads up to 435 MPa. These parameters surpass those of most commercial strain gauges. The measurement results of strain sensitivity and electrical resistance of single-phase equiatomic CoCrFeNi alloy indicate that it is a promising material for strain gauge production.

4. Limitations

The study involved heat treatment of the CoCrFeNi alloy at 1123 K for 24 h. The annealing temperature and duration influence the sample structure and strain sensitivity. More research is required to establish the optimum annealing temperature and duration.

5. Conclusions

In this study, we produced a single-phase equiatomic CoCrFeNi alloy and examined the impact of heat treatment

on its structure, strain sensitivity, and electrical resistivity. The findings are novel in terms of the measured GF values. The main results of this work are as follows:

- 1. According to microstructure analysis, the annealed equiatomic CoCrFeNi high-entropy alloy is a chemically uniform, single-phase disordered solid solution with a finely grained microstructure.
- 2. Cold-rolled foils produced from annealed samples have a 12% greater GF value than those from as-cast alloys.
- 3. The resistivity of the alloys remains linear under elastic stress up to 450 MPa within the ε = 0-0.2% range.

Supplementary materials

No supplementary materials are available.

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None.

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Supervision: V.B. Validation: I.E., E.S. Visualization: V.B.

Writing - original draft: V.B., T.V. Writing - review & editing: V.B.

Conflict of interest

The authors declare no conflict of interest.

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References

- Yeh JW, Chen SK, Lin SJ, Gan JY, Chin TS., Shun TT, Tsau CH, Chang SY. Nanostructured High-Entropy Alloys with Multiple Principal Elements: Novel Alloy Design Concepts and Outcomes. Adv Engin Mater. 2010;6:299-303. doi:10.1002/adem.200300567
- Cantor B. Multicomponent high-entropy Cantor alloys. Prog Mater Sci. 2021;120:100754. doi:10.1016/j.pmatsci.2020.100754
- Uporov SA, Evdokimov IV, Ryltsev RE, Sterkhov EV, Bykov VA, Sidorov VA, Chtchelkatchev NM. High entropy alloys as strain-sensitive materials, Intemetalics. 2024;170:108334. doi:10.1016/j.intermet.2024.108334
- 4. Cantor B, Chang ITH, Knight P, Vincent AJB. Microstructural development in equiatomic multicomponent alloys. Mater Sci Engin A. 2004;375-377:213-218. doi:10.1016/j.msea.2003.10.257
- Jin K, Sales BC, Stocks GM, Samolyuk GD, Daene M, Weber WJ, Zhang Y, Bei H. Tailoring the physical properties of Nibased single-phase equiatomic alloys by modifying the chemical complexity. Sci Rep. 2016;6:20159. doi:10.1038/srep20159
- Kao YF, Chen SK, Chen TJ, Chu PC, Yeh JW, Lin SJ. Electrical, magnetic, and Hall properties of AlxCoCrFeNi high-entropy alloys. J Alloys Compd. 2011;509(5):1607-1614. doi:10.1016/j.jallcom.2010.10.210
- Wu Z, Bei H, Otto F, Pharr GM, George EP. Recovery, recrystallization, grain growth and phase stability of a family of FCC-structured multi-component equiatomic solid solution alloys, Intermetallics. 2014;46:131-140. doi:10.1016/j.intermet.2013.10.024
- Radhakrishnan M, McKinstry M, Chaudhary V, Nartu MSKKY, Mani Krishna KV, Ramanujan RV, Banerjee R, Dahotre NB. Effect of chromium variation on evolution of magnetic properties in laser direct energy additively processed CoCrxFeNi alloys. Scripta Materialia. 2023;226:115269. doi:10.1016/j.scriptamat.2022.115269
- 9. Laplanche G, Gadaud P, Bärsch C, Demtröder K, Reinhart C, Schreuer J, George EP. Elastic moduli and thermal expansion coefficients of medium-entropy subsystems of the CrMnFe-CoNi high-entropy alloy. J Alloys Compd. 2018;746:244-255. doi:10.1016/j.jallcom.2018.02.251
- 10. Sciammarella CA, Sciammarella FM. Strain Gages Introduction to Electrical Strain Gages. In Experimental Mechanics of Solids. Publisher: John Wiley & Sons, Ltd; 2012. 41-73. doi:10.1002/9781119994091.ch3
- 11. Xing L, Yansheng Z. The strain sensitivity coefficient of resistance for an FeMnAlCr antiferromagnetic alloy. J Phys D Appl Phys. 1996;29(3):511-513. doi:10.1088/0022-3727/29/3/004
- 12. Kazi IH, Wild PM, Moore TN, Sayer M. Characterization of sputtered nichrome (Ni-Cr 80/20 wt.%) films for strain gauge applications. Thin Solid Films. 2006;515(4):2602-2606. doi:10.1016/j.tsf.2005.10.077
- 13. Klaas D, Ottermann R, Dencker F, Wurz MC. Development, Characterisation and High-Temperature Suitability of Thin-Film Strain Gauges Directly Deposited with a New Sputter Coating System. Sensors. 2020; 20(11):3294. doi:10.3390/s20113294
- 14. Schmid P, Triendl F, Zarfl C, Schwarz S, Artner W, Schneider M, Schmid U. Influence of the AlN/Pt-ratio on the electro-mechanical properties of multilayered AlN/Pt thin film strain gauges at high temperatures. Sensors Actuators A Phys. 2020;302:111805. doi:10.1016/j.sna.2019.111805
- 15. Cui Y, Li X, Zhang T, Ding W, Yin J. Development of High-Temperature Wire-Grid Thin Film Strain Gauges. Sensors. 2022; 22(19):7595. doi:10.3390/s22197595 Ustinovshikov Y. Phase transformations in alloys of the Ni-Cr system. J Alloys Compd. 2012;543:227-232. doi:10.1016/j.jallcom.2012.05.120