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Review article
Обзорная статья

Improvement of the Cantor alloy's mechanical properties by alloying with niobium and zirconium

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Abstract. Created in 2004, the high-entropy (HEA) five-component Cantor alloy CoCrFeNiMn is still in the focus of attention of researchers in the field of physical materials science due to a good combination of strength and plastic properties, which open up prospects for its use in various high-tech industries. We performed a brief review of recent publications by domestic and foreign researchers on improving the mechanical properties of the Cantor alloy by alloying with niobium and zirconium, which proved themselves well in alloying traditional alloys. Zirconium alloying leads to a lower melting point due to the formation of eutectic with all elements of the Cantor alloy. Alloying with niobium atoms in the range of 0 – 16 at. % ensures the formation of a volume fraction of the Laves phases and σ–phase up to 42 %, which, in turn, is responsible for a fivefold increase in the yield strength from 202 to 1010 MPa. The work on the joint alloying of the Cantor alloy with Zr + Ti + Y_2O_3, Nb + C, Nb + V systems was analyzed. With complex alloying, the mechanical properties are significantly improved. The paper reveals and discusses the physical mechanisms of hardening. Microalloying of 0.2 % Nb alloy with 1.3 % C provides an excellent combination of yield strength (~1096 MPa) and elongation (~12 %) after annealing at 700 °C.

Keywords: alloying, niobium, zirconium, Cantor alloy, hardening

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Улучшение механических свойств сплава Кантора легированием ниобием и цирконием

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Аннотация. Созданный в 2004 году высокоэнтропийный (ВЭС) пятикомпонентный сплав Кантора CoCrFeNiMn по-прежнему находится в фокусе внимания исследователей в области физического материаловедения благодаря хорошему сочетанию прочностных и пластических свойств, которые открывают перспективы его использования в различных наукоемких отраслях промышленности. Выполнен краткий обзор публикаций последних лет отечественных и зарубежных исследователей по улучшению механических свойств сплава Кантора путем легирования ниобием и цирконием, хорошо зарекомендовавшими себя при легировании традиционных сплавов. Легирование цирконием приводит к более низкой температуре плавления из-за образования эвтектики со всеми элементами сплава Кантора. Легирование атомами ниобия в диапазоне 0 – 16 ат. % обеспечивает образование объемной доли фаз Лавеса и σ-фазы до 42 %, что, в свою очередь, ответственно за пятикратное увеличение предела текучести от 202 до 1010 МПа. Проанализированы работы по совместному легированию сплава Кантора системами Zr + Ti + Y_2O_3, Nb + C, Nb + V. При комплексном легировании значительно улучшаются механические свойства. В работе раскрыты и обсуждены физические механизмы упрочнения. Микролегирование 0,2 % Nb сплава с 1,3 % C обеспечивает превосходное сочетание предела текучести (~1096 МПа) и относительного удлинения (~12 %) после отжига при 700 °C.

Ключевые слова: легирование, ниобий, цирконий, сплав Кантора, упрочнение

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Introduction

High-entropy alloys (HEAs), a novel class of metallic materials discovered towards the end of the 20th century, comprise 5 to 6 elements, each at concentrations ranging from 5 to 35 % [1; 2]. These alloys have attracted significant research interest due to their outstanding characteristics, such as high strength, ductility, corrosion resistance, and suitability for extreme temperature conditions, as well as their ease of machining. The potential applications of HEAs span a wide range, from the fabrication of cutting tools and molds to components for the nuclear power and aerospace industries [3 – 8]. According to Scopus, over 30,000 articles on HEAs have been published in the last quarter-century. Among this vast repository of research, the Cantor alloy (CoCrFeNiMn) occupies a prominent position, having been thoroughly investigated for its properties at ambient, high, and low temperatures since 2004 [1; 2]. Remarkably, the Cantor alloy can exhibit an elongation to fracture of approximately 71 % at room temperature, though its yield strength and toughness are relatively low at 215 MPa and 491 MPa, respectively [1; 2]. The practical application of this alloy is somewhat limited by the challenge of balancing strength and ductility, a hurdle that might be overcome through alloying techniques without inducing embrittlement.

The quest to enhance the mechanical properties of HEAs continues to be a key focus for researchers in the field of material science. Review articles [9; 10] have examined two main approaches to address this challenge: firstly, through the development of a nanocrystalline structure and surface hardening via external energy treatments [11; 12], and secondly, through computer modeling to predict high-performance properties, utilizing the CALPHAD software package designed for calculating phase diagrams [13 – 15]. Additionally, the traditional method of alloying with elements well-established in the fabrication of steels and other alloys is also being explored.

The enhancement of both mechanical and functional properties of Cantor HEAs can be achieved through the strategic incorporation of well-investigated elements such as niobium and zirconium [2; 9; 16]. The technique of microalloying with these elements is a well-established practice for hardening conventional steels and alloys. The underlying principles and mechanisms of this hardening process have been the subject of extensive research, particularly within the realm of rail steel from the late last century through the early years of the current century [17 – 19]. Niobium, recognized for its potent carbide- and nitride-forming capabilities, interacts with carbon and nitrogen to generate ultrafine, nano-sized carbides and carbonitrides. Nevertheless, it's important to note that micron-sized particles can adversely affect impact toughness by facilitating a pronounced intergranular fracture mode. Such fractures are among the least energy-intensive and thereby the most hazardous, as the energy absorbed during fracture primarily reflects the properties of the weakened grain boundaries within the polycrystalline structure, rather than the intrinsic characteristics of the metal itself.

During the cooling phase following hot rolling, niobium precipitates as niobium carbide and/or niobium nitride, enhancing the pearlitic structure's hardness (strength) through dispersion hardening and bolstering wear resistance and resilience against internal fatigue failure. Furthermore, niobium plays a crucial role in averting the softening of the heat-affected zone in welds: niobium carbide or nitride consistently forms in the heat-affected zone across a broad temperature spectrum, upon reheating to temperatures at or below the Ac_1 point. Nonetheless, when the niobium concentration falls below 0.001 %, these benefits are not realized to a meaningful degree, leading to no discernible enhancement in the hardness (strength) of the pearlitic structure. Conversely, a niobium content exceeding 0.050 % results in over-intensification of dispersion hardening due to niobium carbide or nitride, rendering the pearlite structure brittle and diminishing the rail's internal fatigue resistance. Therefore, the optimal niobium concentration lies between 0.001 and 0.050 %.

When niobium is incorporated into a complex multi-element alloy like the Cantor alloy, the impact of such alloying additives on the structure and properties of the HEA can only be fully understood through nanoscale investigation using transmission electron microscopy.

The scarcity of publications on this topic means that a unified understanding of how niobium and zirconium influence the alloy's characteristics is still developing. Nevertheless, the existing research efforts dedicated to this area highlight both its importance and practical relevance to the scientific community.

The aim of this article is to briefly review recent studies on the enhancement of mechanical properties in the Cantor alloy through the alloying with niobium and zirconium.

Results and discussion

The role of 5 % zirconium additives in the recrystallization process of the equiatomic Cantor composition was scrutinized in a study [20]. The analysis focused on the cold-rolled alloy subjected to annealing for 30 min at elevated temperatures ranging from 750 to 1125 °C to examine the kinetics of recrystallization. This research aimed to understand the evolution of grain boundaries and grain size from the cast state to the recrystallized condition. It was found that the primary force driving the recrystallization of the dendritic microstructure is the eradication of the dislocation substructure engendered during cold rolling. Alloying with zirconium resulted in more effective solid-solution hardening than the non-alloyed counterpart, maintaining a single-phase HEA structure. The dendritic microstructure transitioned into a fine-grained polycrystalline structure, facilitating CoCrFeNiMn + 5 % Zr HEA's application at cryogenic temperatures.

The zirconium alloying effect was markedly improved by concurrently introducing titanium and yttrium oxide Y_2O_3, each at 1 wt. %, through mechanical alloying followed by plasma sintering [21]. This modified alloy possessed a FCC lattice with a high density of various oxide morphologies (up to $2.01 \cdot 10^{21}$ m^{-3}), contributing to its exceptional mechanical properties. The average grain size was around 130 nm, with oxides forming hexagonal ($YTiO_2$), orthorhombic (Y_2TiO_5), and monoclinic (Ti_2O_3, $Y_2Zr_2O_7$) structures. This high density of oxides and small grain size yielded outstanding microhardness, yield strength, and toughness values of 449 HV, 1309 MPa, and 2231 MPa, respectively. The predominant hardening mechanisms were identified as grain boundary hardening and Orowan hardening.

Comprehensive analysis of how alloying with various elements influences the mechanical properties of the Cantor alloy was presented in [2; 22]. The studies revealed an increase in hardness with the addition of niobium [23] and zirconium [24], attributable to solid-solution hardening and the formation of second phases. Notably, as the niobium content ranged from 0.1 to 0.8 wt. %, hardness linearly escalated to 712 HV, with Laves phases playing a crucial role. Concurrently, an investigation [25] reported a significant increase in yield strength from 1373 to 2473 MPa with a niobium concentration boost from 0 to 5 wt. %. The mechanism of dislocation motion obstruction was identified as a key factor in this enhancement.

In the study documented in [24], $CoCrFeNiZr_x$ alloys with varying zirconium concentrations were synthesized using vacuum-arc melting. A distinct eutectic microstructure was observed in the cast alloy when $x = 0.5$. The research demonstrated that the alloys comprise a FCC solid solution and a Laves C15 phase, appearing in lamellar formations. The crystallographic orientation relationship between these two phases was established. As the volume fraction of the Laves C15 phase increased, the alloys exhibited enhanced strength but showed increased brittleness at room temperature; the mode of fracture transitioned from ductile inter-lamellar to brittle trans-lamellar. However, with elevated test temperatures, fractures tended to be more ductile, indicating that the eutectic microstructure is capable of enduring significant plastic deformation. This characteristic suggests their potential utility in engineering applications at higher temperatures [24]. The impact of alloying is notably amplified when niobium and vanadium (Nb + V) or niobium and carbon (Nb + C) are introduced together [27].

A study [28] delves into the complexities and challenges involved in analyzing hardness changes in CoCrFeNi alloys alloyed with 1 – 4 wt. % zirconium. It was observed that, following annealing at temperatures below 700 °C, the initially formed nanocrystalline grains maintained their size of approximately 10 nm and a hardness of around 500 HV. However, a significant increase in grain size was noted at temperatures of 900 °C and above, reaching up to 250 nm at 900 °C and transitioning to micron-sized grains at 1100 °C. This variation in grain size distribution may provide avenues for developing HEAs with a superior blend of properties by integrating large grains within a fine-grained matrix.

The influence of zirconium on the melting temperature, microstructure, recrystallization, and mechanical properties of the Cantor HEAs is particularly noteworthy, as discussed in article [29]. The research utilized samples prepared through vacuum arc melting of pre-mechanically alloyed powders, followed by 90 % cold rolling and recrystallization annealing at 1143 K. The incorporation of zirconium yielded several benefits, including a faster induction melting process under vacuum conditions, a reduced melting temperature due to zirconium's eutectic formation with the Cantor alloy elements, improved chemical homogeneity, and enhanced mechanical properties of the recrystallized grains. The zirconium-altered HEA exhibited a higher recrystallization temperature and reduced grain size post-recrystallization, which contributed to increased hardness and strength of the alloy.

The beneficial role of niobium microalloying in a carbon-containing Cantor alloy was explored in article [30]. Such fine-grained, carbon-alloyed HEAs demonstrated an optimal mix of yield strength and ductility. Nonetheless, these carbon-infused HEAs are prone to decomposing into intermetallic compounds under intermediate temperatures, presenting a challenge to their structural stability and performance. The integration of a mere 0.2 % niobium into the CoCrFeMnNi – 1.3 % C (Nb – HEA) alloy markedly enhances mechanical performance at room temperature while averting thermal decomposition at intermediate temperatures. Niobium's microalloying role in carbon-enriched high-entropy alloys is crucial, facilitating the release of NbC carbides at temperatures between 700 and 900 °C, thus inducing hardening. The Nb – HEA alloy, particularly after annealing at 700 °C for 1 h, exhibits an impressive synergy of yield strength (approximately 1096 MPa) and relative elongation (approximately 12 %). Furthermore, niobium microalloying curtails the disintegration of the FCC matrix at intermediate temperatures (500 °C), significantly reducing the emergence of brittle σ-phase, while restraining the proliferation of L10 and BCC/B2 phases.

The CALPHAD software suite serves as a predictive tool for behavior of the Cantor's HEA during alloying processes [23]. Through computer-aided thermodynamic analyses, a pseudo-eutectic binary $CoCrFeNiNb_x$ alloy series (with x values of 0.10, 0.25, 0.50 and 0.80) was devised. Experimental findings reveal that these eutectic alloys comprise a ductile face-centered cubic (FCC) phase alongside a solid Laves phase, characterized by a fine lamellar structure, thereby endowing the alloys with superior mechanical attributes in terms of both plasticity and strength. For the $CoCrFeNiNb_{0.5}$ variant, tensile strength under compression and strain-to-fracture metrics exceeds 2300 MPa and 23.6 %, respectively. Informed by CALPHAD projections [26], a septenary eutectic high-entropy alloy (comprising Fe, Ni, Cr, V, Co, Mn, and Nb) was synthesized via a melting approach. The configurational entropy calculated for the dual-phase microstructure qualifies the alloy as a high-entropy alloy. Notably, when niobium concentration surpasses 9.7 wt. %, the microstructural paradigm shifts from pre-eutectic with primary FCC to hypereutectic with dominant Laves phase.

The structural phase states and hardness of two distinct non-equiatomic HEAs, namely Cantor and Cantor + NbC, were meticulously explored in [27]. The empirical evidence aligns with CALPHAD-based theoretical predictions, suggesting the presence of two solid solutions characterized by high entropy and a FCC crystalline structure post-centrifugal casting. Microscopic examinations and hardness assessments detected minimal structural variance across the thickness of both alloys, depicting a dendritic configuration with iron and manganese segregating within dendritic zones, whilst inter-dendritic spaces concentrated cobalt, chromium, and nickel. Niobium-rich nano-precipitates exhibiting spherical and oval shapes were discernible in interdendritic areas. Differential thermal analysis did not register any peak up to the melting point, indicating the solid solution structures' high temperature resilience.

The beneficial impact of Laves phases on the mechanical properties of HEAs has been underscored across various studies. A particular investigation [31] traced the microstructural evolution and mechanical performance of $(CoCrFeNiMn)_{100-x}Nb_x$; $0 \leq x \leq 16$ at. % under compression. This study demonstrates that the volumetric fraction of secondary phases (Laves and σ-phases) escalated from 0 to 42 %, correlating with a yield strength increase from 202 to 1010 MPa.

The lamellar structures have transient mechanical properties. Although the mechanical properties deteriorate as these structures degrade after annealing at 900 °C, HCEs from $CoCrFeNiNb_x$ retain good mechanical properties.

Conclusions

This brief review synthesizes the findings from recent domestic and international research articles on modification of the Cantor alloy through alloying with zirconium, niobium, and complex alloying using systems such as Nb + V, Nb + C, Zr + Ti + V_2O. The discussions in these studies primarily focus on the physical mechanisms of hardening induced by these alloying processes.

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M. O. Efimov – analysis of articles on the combined effect of niobium, zirconium and other elements on the Cantor alloy's properties.
I. A. Panchenko – analysis of influence of niobium and zirconium on the Cantor alloy's mechanical properties.
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