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High-entropy alloys. A new concept for the design of innovative structural materials

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Високоентропійні сплави. Нова концепція проектування інноваційних конструкційних матеріалів

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Abstract. Modern technologies require state-of-the-art materials that meet their conditions, regardless of operating conditions. Alloys with high entropy can replace traditional materials, work under impacts, dynamic loads, elevated temperatures, etc. These alloys are used for the manufacture of tools, molds, dies, mold casting in parts that require high strength, resistance to oxidation and wear, can also be used in environments with high corrosion resistance parameters (plumbing, marine conditions), in aggressive conditions and in the chemical industry. High entropy alloys are quite easy to investigate and control, and can be obtained by the same methods as traditional alloys, such as: casting, rapid melt quenching, film sputtering, electrolysis, and mechanical alloying. Electroslag remelting (ESD) can greatly improve the purity, hardening structure, and transverse mechanical properties of steel. However, the increasing demands on the mechanical properties of steel are prompting metallurgists to make more efforts to eliminate defects in steel microstructures such as shrinkage and segregation. The combination of directional crystallization technology with electroslag melting technology effectively eliminates macrosegregation in the cast ingot through a shallow molten metal bath controlled by directional crystallization. Increasing the strength of alloys can be achieved either by alloying a solid solution (elements in the internodes) or by isolating the solidification phases or artificially introducing microparticles. Curing phases (carbides, nitrides, carbonitrides, intermetals) can be endogenous (formed from elements introduced into the melt in a liquid state or during its solidification and subsequent cooling) or exogenous (usually introduced into the melt just before crystallization begins, and there is also an increase in size and deterioration in the distribution of solidification phases).

Keywords: high-entropy alloys, strength, hardening phases, electroslag remelting.

Анотація. Сучасні технології потребують сучасних матеріалів, які відповідають їхнім умовам, незалежно від умов експлуатації. Сплави з високою ентропією можуть замінити традиційні матеріали, працювати під ударами, динамічними навантаженнями, підвищеними температурами тощо. Ці сплави використовуються для виготовлення інструментів, форм, штампів, лиття у деталях, що потребують високої міцності, стійкості до окиснення та зношування, а також можуть застосовуватися в середовищах з високими параметрами стійкості до корозії (сантехніка, морські умови), у агресивних умовах та в хімічній промисловості. Сплави з високою ентропією досить легко досліджувати та контролювати, і їх можна отримати тими ж методами, що й традиційні сплави, такими як: лиття, швидке загартування плавом, розпилення плівки, електроліз і механічне легування. Електрошлакове переплавлення (ESD) може значно покращити чистоту, структуру загартування та поперечні механічні властивості сталі. Однак зростаючі вимоги до механічних властивостей сталі спонукають металургів докладати більше зусиль для усунення дефектів сталевих мікроструктур, таких як усадка та сегрегація. Поєднання технології напрямленої кристалізації з технологією електрошлакового переплавлення ефективно усуває макросегрегацію в литому злитку через неглибоку ванну з розплавленим ме-



талом, контролювану напрямленою кристалізацією. Підвищення міцності сплавів можна досягти або шляхом легування твердого розчину (елементів у міжвузлах), або шляхом ізоляції фаз затвердіння або штучного введення мікрочастинок. Фази затвердіння (карбіди, нітриди, карбонітриди, інтерметали) можуть бути ендогенними (утвореними з елементів, введених у розплав у рідкому стані або під час його затвердіння та подальшого охолодження) або екзогенними (зазвичай вводяться в розплав безпосередньо перед початком кристалізації, а також спостерігається збільшення розміру та погіршення розподілу фаз затвердіння.

Ключові слова: сплави з високою ентропією, міцність, фази загартування, переплавка електршлаку.

Introduction

Alloying has long been used to give materials the desired properties. It usually involves the addition of small amounts of secondary elements to the primary element. However, in the last decade and a half, a new alloying strategy has become widespread, involving the combination of several basic elements in high concentrations to create new materials called high-entropy alloys [1]. The multidimensional compositional space that can be explored with this approach is virtually limitless, and only tiny areas have been explored [2]. The materials science community, which is no longer safe at the corners and edges of triple phase diagrams, now finds itself in uncharted, hyperdimensional territory that is difficult to imagine, difficult to visualize, and difficult to systematically explore [3]. The concept of huge compositions and microstructures associated with alloys with several basic elements did not lose its relevance in the first 12 years. Significant progress has been made, and it continues to motivate new research questions and inspire new important scientific topics. The combinatorial enormity of this idea also poses the greatest technical challenge that this industry offers. Of the literally hundreds of millions of possible combinations of elements, barely a hundred have been studied so far.

Review of scientific information

Due to the multiplicity of base elements and multiple effects, they have different organizational structures and unique properties compared to traditional alloys, which opens up unlimited development potential and promising applications of wind farms. For almost 30 years of development, the methods for obtaining and studying wind farms have expanded significantly, the systems have been optimized, and the scope of their application has expanded. This article provides a comprehensive overview of the development of manufacturing processes, including wind farms in the form of blocks, coatings, wire, powders, and additive technologies. In addition, a number of research results on the critical properties of wind farms, such as mechanical properties, corrosion resistance, wear resistance and oxidation at high temperatures, as well as new functional properties such as radiation resistance, hydrogen capability and biocompatibility, etc., have been summarized [1].

Based on research on intermetallic structural materials [4, 5] and massive metallic glasses [6], the concept of "high-entropy alloys (HEA)" or "multiple-core alloys (MPEA)" was introduced in two papers independently published by J.W. Yeh et al. [7, 8] and

B. Cantor et al. [9] in 2004. Since then, there has been an explosive development in the field of materials. According to the composition, HEA are defined as alloys consisting of at least 5 elements and a maximum of 13 elements. To expand the scope of alloy development, the molar fractions of each major element were $>5\%$ and $<35\%$ [10].

H13 steel is a prime example of Cr-Mo-Si-V hot-working tool steels known for their extreme strength, resistant red hardness, and extreme resistance to wear and thermal stress [11]. Its wide application in the manufacture of molds and dies can be explained by these exceptional properties. Traditionally, molds are made using casting and machining methods. However, these traditional production methods are characterized by low production rates and significant limitations in design flexibility. In recent years, laser powder cladding (LPBF), due to its unique advantages such as geometric design flexibility, the ability to create complex structures, which contributes to cost-effective product customization and provides excellent mechanical properties, has become a promising method for manufacturing metal components and is widely used in the aerospace, medical, automotive, and mold manufacturing industries [11].

Tool steels must not only provide wear resistance, but also have good toughness, which can guarantee a long service life under wear conditions [13, 14]. For example, tools for rolling panel machines, usually made of tool steels with a high chromium content [15], have good mechanical properties characterized by impact toughness as well as high wear resistance [16]. The main reason is that when a rolling tool is used to roll rocks, it undergoes abrasive wear under high load, and a large amount of heat is released due to instantaneous friction, resulting in a rapid increase in the surface temperature of the tool [17]. After rotation, the surface of the tool is immediately cooled by spraying water. In this way, the tool not only withstands wear and tear [18], but also withstands cold and hot fatigue. However, for some tool steels with a specific composition, wear resistance and toughness are contradictory. Under the condition of high wear resistance, the impact strength of steels is usually low [19]. Therefore, it is necessary to balance impact strength and wear resistance in order to improve tool life [25]. Broadly speaking, there are many methods to improve the conflicting mechanical properties of steel, such as ultra-pure cleaning, heat treatment, large forging ratio, etc. [20, 21]. However, on a technical level, tool and stamped steel for cutting tools has reached a certain limit. Therefore, the development of a new

and effective method that can improve the impact strength and wear resistance of steels is of high practical importance and industrial application [22]. Traditional methods for improving the complex mechanical properties of steel include adding metal elements to the steel die for alloying and optimizing the composition, developing new heat treatment processes, and developing new molding manufacturing processes. However, it is difficult to simultaneously increase the strength and strength of steel with these traditional methods [22]. The incorporation of trace ceramic particles into the steel matrix is an effective method to further improve the mechanical properties of steel due to its low cost and high performance [23]. One of the most attractive methods for increasing the strength of tool steel is to add ceramic rebar to a steel-based die, which has a low density and a homogeneous microstructure with better mechanical properties. Metal matrix composites have been developed due to their attractive properties such as high specific strength, modulus, thermal resistance and excellent wear resistance. Solid ceramic particles such as TiC and TiB₂ are used to improve mechanical properties, temperature stability, and wear in steel composites. TiC is well known as a reinforcement in steel composites due to its desirable characteristics such as high melting point, low density, high modulus of elasticity, and good wettability with iron and steel matrices. Stable carbides coarsen more slowly than cementite and are therefore much more efficient than cementite at higher temperatures. Together with the solid martensitic matrix, the addition of TiC, which is thermodynamically stable in contact with the steel matrix [22, 25-27], there is a significant increase in stiffness, hardness, and wear resistance.

The paper [28] presents the results of a systematic study of the effect of the Cr/V ratio (0.2, 0.4, 0.6, 0.8 and 1.0) on the microstructure and tensile strength of high-vanadium high-speed steel. The results show that lower Cr/V ratios contribute to the formation of coarse block carbides of the MC type, which are gradually crushed and undergo spheroidization with an increase in the Cr/V ratio. The evolution of the matrix phase reveals a non-monotonic trend towards austenite content, peaking at approximately 40% for a Cr/V ratio of 0.6, while the martensite content is inversely related.

[29] The effect of the axial static magnetic field (ASMP) on carbides and mechanical properties of stamped H13 steel obtained by electroslag melting was investigated. An optical microscope (OM) and a scanning electron microscope (SEM) were used to analyze the solidification structure and morphology of carbides of electroslag melting ingots (ESD). To detect inclusions in ESP H13 ingots, the FEI Aspek Explorer device was used. Compared to conventional ESP, the application of ASMP in this study made the molten metal bath more superficial and gentle, resulting in a reduction in local solidification time and uniform distribution of solute atoms, which

reduced the degree of segregation of elements and further inhibited the formation of primary carbides. The magnetically controlled electroslag remelting (MC-ESR) process not only crushed the structure of dendrites (which tended to grow parallel to the ingot axis) and carbides, but also improved the inclusion removal efficiency and mechanical properties of H13 steel ESP ingots.

Authors [30, 31] studies of microstructure and carbides in cast austenitic mold steel produced by traditional electroslag remelting (ESR) and continuous directional electroslag remelting (ESR-CDS) methods have been performed. In addition, the growth pattern of carbides was also considered. A combination of optical microscopy (OM) and scanning electron microscopy (SEM) was used to characterize the microstructure and carbides. Segregation was analyzed using an initial position analyzer (OPA) and electron probe microassay (EPMA). Electrolytically extracted carbides were analyzed using SEM and X-ray diffraction (XRD) to determine their three-dimensional microstructure and composition. The microstructure of the steel consisted of an austenitic matrix and primary carbides of the V₈C₇ and Mo₂C types. Compared to traditional ESR, ESR-CDS contributed to the formation of a finer microstructure in cast steel, a smaller amount and smaller size of carbides in melted steel. Meanwhile, the segregation of alloying elements was reduced with ESR-CDS. Enrichment with carbide-forming elements was reduced due to directional solidification of ESR, resulting in a change in the morphology of V-rich carbides from rod-shaped to lamellar shape. The hardness and impact strength of the melted ingot (made with ESR-CDS) after heat treatment (solution temperature 1180°C for 2 hours, aging temperature 720°C for 2 hours) increased by 3-5 HRC and 4-6 J/cm², respectively, compared to the results obtained with conventional ESR.

A thorough study is presented in the publication [32]. To analyze the curing and deposition behavior of primary carbides in H13 steel, the size, morphology, distribution, and type of carbides from the cooling edge to the center of H13 steel are studied using a scanning electron microscope (SEM), X-ray diffractometer, and Thermo-Calc thermodynamic software, and both carbide formation time under equilibrium conditions and non-equilibrium solidification are discussed. The results show that the primary carbides are distributed in the final curing of the steel, mainly including MC type V-Ti PC, V PC and M₇C₃ type Mo-Cr PC. From edge to center, the average area of primary carbide increases by 620.22 μm², with an increase of 3.53%. From the edge to the ¼ position, there are mainly V-rich and Mo-Cr carbides; at position 1/4, carbides rich in V and Mo-Cr are interconnected; from position 1/4 to the center, there are three types of interrelated V-Ti PC, V-rich PC, and Mo-Cr PC carbides. Oxides promote MC-type precipitation, while sulfides promote M₇C₃-type precipitation. Thermodynamics shows that the pri-

mary carbide MC is not formed in equilibrium solidification, but is precipitated in a non-equilibrium state with a precipitation temperature of 1108°C at a solidification fraction of 0.9987.

Thermodynamic analysis of carbide formation with the participation of steel components H13 and D2.

UNS T30402 Type D2 steel is intended for the manufacture of tools used for cold processing of metals and other materials. It is characterized by a high content of carbon and chromium, has high

resistance to abrasion. The steel is used for the manufacture of punching dies, mandrels, rolling dies, tools with high mechanical strength and toughness.

UNS T20813 Type H13 is intended for the manufacture of tools used for processing metals under pressure at temperatures above 300°C. The steel is used for the manufacture of oil- and air-cooled mandrels and mandrels of extrusion presses, molds for injection molding.

Table 1 - Chemical composition of UNS T20813 Type H13 steel and the closest analogue 4X5MF1C.

Chemical element, %	UNS T20813 Type H13		4X5MF1C	
	Min.	Max.	Min.	Max.
C	0,32	0,45	0,37	0,44
Mn	0,20	0,60	0,20	0,50
Si	0,80	1,25	0,90	1,20
Cr	4,75	5,50	4,50	5,50
V	0,80	1,20	0,80	1,10
Mo	1,10	1,75	1,20	1,50
Ni		0,30	-	0,30
W		-	-	0,20
Cu		0,25	-	0,30
Ti		-	-	0,03
P	-	0,030	-	0,030
S	-	0,030	-	0,030
Fe	main element		main element	

Table 2 - Chemical composition of UNS T30402 Type D2 and the closest analogue X12MF.

Chemical element, %	UNS T30402 Type D2		X12MF	
	Min.	Max.	Min.	Max.
C	1,40	1,60	1,45	1,65
Mn	0,10	0,60	0,15	0,45
Si	0,10	0,60	0,10	0,40
Cr	11,0	13,0	11,0	12,5
V	0,50	1,10	0,15	0,30
Mo	0,70	1,20	0,40	0,60
Ni	-	0,30	-	0,35
Ti	-	-	-	0,03
Cu	-	0,25	-	0,30
W	-	-	-	0,20
P	-	0,030	-	0,030
S	-	0,030	-	0,030
Fe	main element		main element	

According to the chemical composition given in Tables 1 and 2, the main carbide-forming elements are: V, Cr, Mo, Mn, Fe. The sequence and intensity of the formation of carbides of these elements are determined by the magnitude of their chemical affinity for carbon, which is clearly confirmed by the mutual arrangement of the corresponding temperature dependences on the Ellingham diagram (Fig. 1, 2).

The results showed that the primary carbides in

cast H13 steel are mainly composed of Cr, Mo, V and Ti, and there are four types of primary carbides in the interdendritic zones of H13 steel: M₂C type, enriched in Mo-Cr; eutectic M₂C type, enriched in Mo-Cr; MC type, enriched in V, and MC type, enriched in V, with Ti and N.

In H13 steel, primary carbides significantly affect the tool life. The precipitation of primary carbides reduces the solubility of Cr, Mo and V in the solid

state in the matrix, thereby reducing the precipitation of secondary carbides during tempering and affecting the homogeneity of the microstructure. Uniformly dispersed nanoparticles are vital for the secondary strengthening of H13 steel. However, due to the high

temperature stability of primary carbides, they cannot be removed during heat treatment. Large primary carbides can become sources of cracks, leading to thermal fatigue failure and reduced tool life.

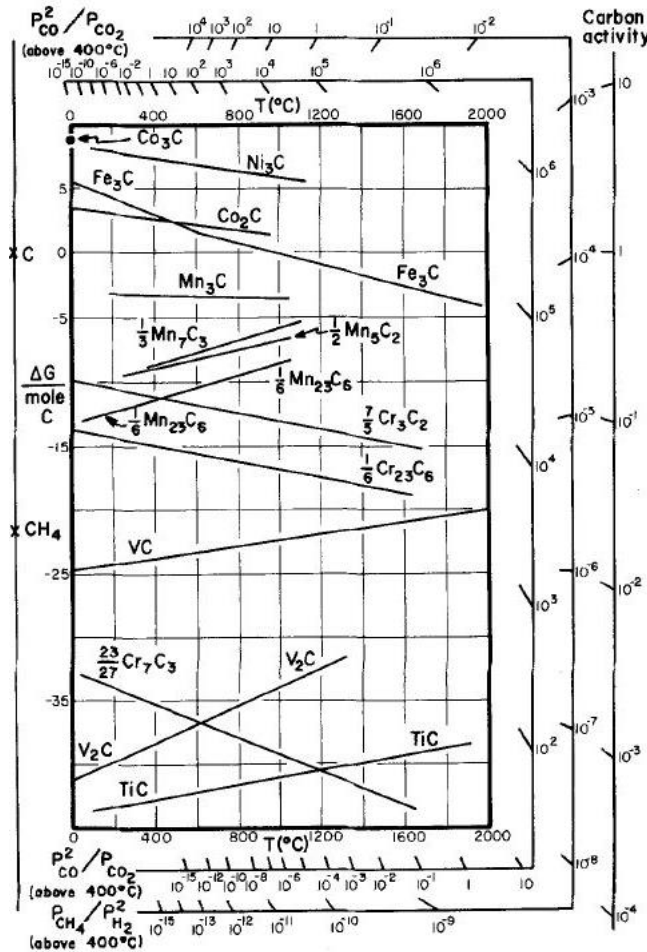


Figure 1 - Ellingham diagram for carbides of the first transition series.

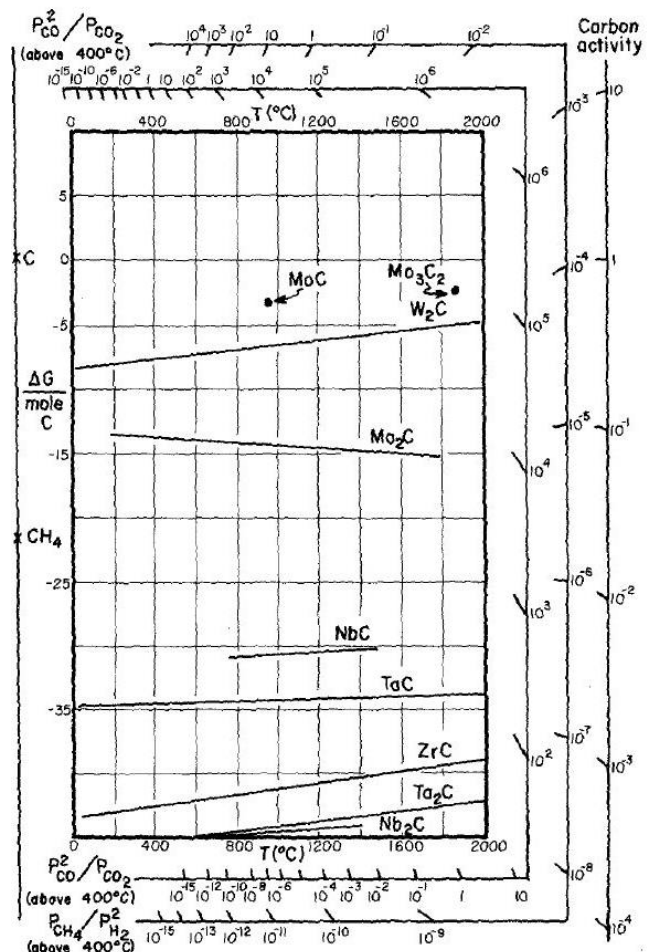


Figure 2 - Ellingham diagram for second and third transition series carbides.

Conclusions

At present, high-entropy alloys are a promising direction for the development of innovative materials. Modern technologies require the latest materials that will meet their conditions, regardless of the operating environment.

High-entropy alloys can replace traditional materials, they can work under shock, dynamic loads, elevated temperatures, etc.

High entropy alloys are suitable for the manufacture of tools, molds, dies, casting molds in parts that require high strength, oxidation resistance and wear resistance, can also be used in environments where high corrosion resistance parameters are required (plumbing, marine environments), in aggressive environments and in the chemical industry. High-entropy alloys are quite simple to investigate and control, they can be obtained by the same methods as traditional alloys, i.e.: casting, rapid melt quenching, film sputtering, electrolysis and mechanical al-

loying.

Electroslag remelting (ESD) can greatly improve the purity, curing structure, and transverse mechanical properties of steel. However, increasing demands on the mechanical properties of steel are prompting metallurgists to put more effort into eliminating steel microstructure defects such as shrinkage and segregation. The combination of directional crystallization technology with electroslag melting technology effectively eliminates macrosegregation in the cast ingot due to a shallow bath of molten metal controlled by directed crystallization.

An increase in the strength of alloys can be realized either by alloying a solid solution (elements in the internodes) or by isolating hardening phases or artificial introduction of microparticles. Hardening phases (carbides, nitrides, carbonitrides, intermetallics) can be endogenous (formed from elements introduced into the melt in a liquid state or during its solidification and subsequent cooling), or exogenous

(usually introduced into the melt immediately before the start of crystallization, and there is also an increase in the size and deterioration of the distribution of hardening phases. phases is 30-70% mass.

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