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Selection of inoculant additives for modifying nickel alloys

Heat-resistant nickel alloys are widely used in the production of castings for aircraft and industrial gas turbine engines. Structural factors are the main determinants of the performance properties of cast nickel alloys. The main disadvantage of castings obtained from these alloys is the coarse-crystalline structure, uneven grain size and columnar crystals in the cross-section. Therefore, the creation of an optimal alloy structure is an important condition for obtaining high properties and ensuring the increased operability of cast parts. Obtaining a fine-grained structure has a beneficial effect on the level of mechanical and operational properties of cast metal. The most promising way to create such a structure is to introduce a small number of additives into the melt that cause heterogeneous formation of crystal nuclei, i.e. modification of the melt with dispersed particles of refractory elements and inocular compounds. To select the type of inocular particles required to initiate crystallization of a particular phase, it is necessary to have a set of data that allows one to form a theoretical understanding of the principles of such a choice. The paper provides a rationale for the selection of the type of particles of inoculators capable of causing the process of artificial changes in the structure of cast metal. For a heat-resistant nickel alloy, the use of refractory particles of ultra-dispersed titanium carbo nitride powder as inoculators are the most effective. When introduced into the melt 0.025 wt. % of such particles, a fine-grained structure of the alloy is obtained, and its ductility in comparison with the unmodified one is more than doubled.

Keywords: high-temperature nickel alloy, melt, modification, crystallization, inoculators, particles, properties, structure.

Introduction

Heat-resistant nickel alloys of various alloying degrees are widely used in many industries. They are mainly used in the production of castings of the most critical, highly loaded parts for aircraft and industrial gas turbines of engines [1, 2].

Heat-resistant cast nickel alloys are complex multi-component hetero phase systems. Structural factors, along with chemical composition, are the main factors that determine such properties of nickel alloys as ductility, heat resistance, fatigue resistance, and others [3]. The disadvantage of castings obtained from these alloys is a coarse-crystalline structure, uneven grain size and columnar crystals in the cross-section. Therefore, the creation of an optimal structure for a given alloy is an important condition for obtaining the required properties and ensuring increased material performance.

The most versatile tool that has a beneficial effect on the level of mechanical and operational properties of cast metal is to obtain a fine-grained structure [2, 3]. The greatest effect of dispersion of the primary structure should be expected when it is formed as fine equiaxed grains. To obtain such a structure, certain conditions must be created. One of these conditions is the creation of the entire volume of the melt of a minimum temperature gradient, which would be distributed within the range of crystallization of the alloy. In this case, the nucleation of crystals would occur simultaneously throughout the entire volume of the bath. The most promising way to create such crystallization centers is to introduce into the melt a small number of additives that cause heterogeneous formation of crystal nuclei, i.e. modification of the melt with dispersed particles of refractory elements and inocular compounds. This modification method is a universal way of controlling the crystal structure of cast metal [4–6].

At the same time, to select the type of inocular particles required to initiate the crystallization of a particular phase, it is necessary to have a set of data that allows one to form a theoretical understanding of the principles of such a choice. The available information is extremely contradictory. Thus, J.V. Wood [7] showed the universality of the action of only two types of particles: TiC and ZrB_2 in steels crystallizing through α- and γ- lattices. As established by J. Campbell and J.W. Bannister [8], TiC refines grain in steel with α-grid, and there are no reliable inoculators for phases with a γ-grid. G.S. Ershov and V.A. Chernyakov [9] concluded that only TiN, ZrN, and ZrB_2 can serve as stable inoculators of steel.

Ahlroth & Kettunen [10] found that as a result of the introduction of 0.3... 0.4 % of refractory metal particles (molybdenum, niobium, tungsten) into nickel alloys with a size of 200–400 microns, the metal structure changed significantly, which led to an increase in the plastic properties of the resulting castings. In [11], a similar positive effect was obtained when modifying chromium-nickel stainless steel with refractory metals.

Using the available factual material, it is possible to formulate the basic requirements for inoculating particles. They must have:

– high thermodynamic stability in the melt;
– the highest possible electrical conductivity compared to the melt;
– possibility to differ less from the melt in terms of density.

In connection with the above, the work posed substantiating the choice of the type of modifier particles capable of causing the process of artificial changes in the structure and properties of nickel alloy castings.

Experimental

Alloy Kh10N60K10V10Yu5T3M2B, which is the most prominent representative of the family of high-alloy heat-resistant nickel alloys, widely used for manufacturing parts for aviation equipment [2], was chosen as the object of research. The casting of the test ingots was carried out by vacuum induction melting, in ceramic crucibles, on a U117–7 remelting unit. The chemical composition of the cast metal is shown in Table 1.

Table 1

Alloy composition Kh10N60K10V10Yu5T3M2B by main alloying elements

Composition	Content of elements,%							
	C	Cr	Co	Al	Ti	Mo	W	Nb
Requirements for Specifications	0.13–0.19	8.0–9.0	9.0–10.1	5.1–5.7	2.0–2.6	1.2–1.7	9.5–10.1	0.8–1.1
Investigated alloy	0.18	8.5	9.9	5.6	2.4	1.6	9.8	0.9

The experiments were carried out on cast samples of alloy Kh10N60K10V10Yu5T3M2B with various additions of refractory particles in amounts used in the practice of modification. For modification, we used plasma-chemical synthesis powders with a dispersion of about 100 nm. The introduction of particles into the liquid metal was carried out using briquettes as tablets. They were obtained by mixing powder components followed by pressing at a pressure of 10–15 t/cm^2 on a PG-476 hydraulic press in a specialized press mold of the size corresponding to the tablet, ensuring its dissolution in the melt for 20–40 seconds.

The ingots were cast using the following parameters:

– Operating frequency of the generator — 25650 Hz;
– Residual pressure in the melting chamber — 10–2 mm Hg;
– Heating temperature of the melt — 1700^{o}C;
– Holding time at a given temperature (power 5 kW) — 5 min;
– Temperature of modifier input — 1650^{o}C;
– Holding time at a given temperature — 3 min;
– Cooling of the melt (power 0 kW) — 10 min.

The macrostructure of the alloy was investigated on thin sections after etching in a solution of ferric chloride and a Marble reagent. A Carl Zeiss Axio Observer Alm optical microscope was used for metallographic analysis.

Tensile tests were carried out under the requirements of GOST 1497–84.

Results and Discussion

The choice of inoculars was made based on their resistance to dissolution in the melt, and then their electrical conductivity and density were taken into account.

Analysis of the heterogenizing ability of refractory compounds of the putative modifiers shows that, from the point of view of resistance to chemical interaction and dissolution in the melt, they do not all meet the requirements. Thus, borides are not stable in nickel and its alloys because of the high diffusion mobility of boron and the ability to form low-melting boron nickelides. Oxides are the most stable in terms of resistance to chemical interaction and dissolution, but they are poorly wetted by nickel alloys and can only be introduced together with thermodynamically active elements (Ca, Ba, etc.), the use of which is fraught with great difficulties. Therefore, the greatest preference when choosing modifiers, especially for high-temperature alloys, is given to inoculators in the form of nitrides, carbides and some oxides that are well wetted by melts [4, 7, 12–14] (Table 2).

Table 2

Contact angles of wetting θ in the system refractory compound — Ni *

Compound	θ^*	T, °C	Environ
TiC	38/62/30	1 500/1 550/1 450	vacuum
ZrC	24/32/30	1 400/1 500/1 550	vacuum
VC	~0/-/17	1 450/-/1 380	vacuum
NbC	~0/-/18	1 450/-/1 380	vacuum
TaC	~0/-/16	1 400/-/1 380	vacuum
HfC	-/-/28	-/-/1 450	argon
TiN	70/-/98	1 550/-/1 450	argon
Si_3N_4	90/-/105	1 435/-/1 500	argon
ZrN	-/10/72	-/1 500/1 550	argon
TiB_2	64/-/39	1 480/-/1 550	helium
ZrB_2	55/-/42	1 500/-/1 500	vacuum
CrB_2	40/30/20	1 500/1 460/1 500	vacuum
Al_2O_3	128/-/-	1 500/-/-	vacuum
ZrO	118/-/-	1 500/-/-	vacuum
TiO_2	120/57/-	1 500/1 480	helium/vacuum
Cr_2O_3	0/56/-	1 400/1 500	argon/vacuum

* Work data are separated with a slash [12], [13], and [14]

The phase diagrams of Ni-C-Me (carbide-forming element) studied by H.J. Goldschmit [15] indicate that all the most stable carbides (TiC, ZrC, HfC, NbC) exhibit polymorphism in low temperatures and carbon concentrations region, can be present in the of bcc and hcp modifications, and in high temperatures and carbon concentrations region, all carbides have an fcc lattice and can serve as crystallization centers.

Polythermal section of the Ni TiC system, investigated by V.N. Eremenko, showed that it is quasi-binary and has the form of a simple diagram with a eutectic [16]. The maximum solubility of TiC in nickel occurs at a eutectic temperature of 1280°C and is 6.2 %. The solubility of carbides in liquid nickel alloys decreases in the order TiC → NbC → ZrC → HfC, but only at moderate temperatures. These carbides, as well as other interstitial phases, are characterized by a tendency to deviate from the stoichiometric composition with a deficit in carbon content, which increases the decomposition temperature of carbide systems. The deviation decreases in the following sequence [15]: $TiC_{0.28} \rightarrow ZrC_{0.28} \rightarrow HfC_{0.5} \rightarrow NbC_{0.7}$.

Deviations from stoichiometry play an important role in the kinetics of diffusion, decarburization, dissolution and oxidation processes. The vacancies in carbides can be filled with atoms of components, for example N and O. Carbonitrides, and especially oxycarbonitrides, are more stable in metal melts. However, it is known that in cermets of the MeOx-MeN type, with an increase in the proportion of MeO, the electrical resistance increases, i.e. the proportion of free electrons decreases and the wettability deteriorates. The contact angles θ of carbides with nickel alloys are low, nitrides are wetted worse, and oxides are poorly wetted (Table 2). Improvement of the wetting of refractory compounds with liquid metals occurs after a certain time of their contact due to ion-exchange reactions and mutual diffusion.

Nitrides are more stable than carbides [4, 15, 16]. At the same time, the use of nitrides for modification can lead to an increase in the nitrogen concentration in the melt and the formation of eutectic nitrides along the grain boundaries. However, a moderate increase in the nitrogen content (0.003 %) has a modifying effect on the structure of heat-resistant nickel alloys. With a further increase in its concentration, the plasticity of alloys in which there are no primary MeC carbides deteriorates, and in those alloys in which they are present, an increase in the nitrogen content worsens their morphology, which also has a negative effect on the properties of the metal.

Usually, when modifying, coarse nitride powders with a particle size of 10–200 μm are used in an amount of 0.1–0.2 % of the melt mass [4]. The dissolution of such a quantity of nitrides causes a significant increase in the nitrogen concentration in the melt and contributes to the accumulation of nitrides in the circulating charge. The use of ultrafine powders (UDP) by an order of magnitude reduces the amount of added additive (0.01–0.02 % of the melt mass) and reduces the risk of nitride accumulation [6].

Considering the above and comparing the properties of the main refractory compounds, it is possible to choose a number of inoculant additives that most fully satisfy the requirements imposed on them (Table 3).

Table 3

List of compounds that best meet the selection criteria for inoculators

Compound	Density at 1550 °C, 10^{-3} kg/cm^3	Specific electrical resistance at 1550 °C, μOhm·cm	Literary source
TiB_2	4.43	105	13
ZrB_2	5.91	66	13
WC	15.49	31	13
TiC	4.73	147	13
ZrC	6.44	175	14
NbC	7.29	120	13
HfC	12.3	140	13
TaC	13.89	112	14
TiN	5.33	132	12
ZrN	7.06	126	12
HfN	12.96	122	12
TiCN	4.9	-	17
VN	6.04	-	13
TaN	14.1	138	14
VC	5.36	-	14

Examining data from Tables 2, 3, we conclude that for the modification of nickel alloys; it is most rational to use carbides and nitrides of refractory compounds, primarily titanium as inoculants. An even greater effect can be expected from titanium carbonitride, which has the highest stability in metal melts [17, 18]. Consequently, for the castings of experimental samples, titanium carbides, nitrides, and carbonitrides were chosen as inoculant particles during modification.

The amount of refractory particles in the modifier, which ensures the optimal structure and properties of the cast metal, depends on many factors: the chemical state of the alloy, technological parameters of melting and pouring, etc., as a result of which these parameters can be determined for a specific case only experimentally.

Castings from the alloy under study have extremely low plasticity values. So, the values of the relative elongation of the cast metal obtained by vacuum induction melting are usually in the range of 3.0–3.7 %, which in many cases does not meet the production requirements [2].

The study of the influence of the type and amount of the selected particles-inoculators on the elongation (Table 4) confirmed the validity of the theoretical conclusions.

Table 4

Influence of the type and amount of selected particles on the elongation of the alloy

Inoculator	Average value of the relative elongation depending on the number of particles (weight),%				
	0.01	0.025	0.05	0.075	0.1
TiN	4.6	6.2	4.8	4.6	4.1
TiC	4.4	5.5	4.5	4.2	3.9
TiCN	4.9	7.6	6.1	4.7	4.5

The use of ultrafine powder of titanium carbonitride as particles — inoculators is the most effective. Introducing 0.025 wt.% of such particles into the melt increases the plasticity of the alloy by more than 2 times compared to unmodified, while the results are much worse when other particles are used.

Metallographic studies show that modification of the alloy Kh10N60K10V10Yu5T3M2B with titanium carbonitride manifests itself in a significant refinement of the macrograins of the alloy (Figure 1).

a) b)

a — initial; b — modified

Figure 1. Macrostructure of a microsection of alloy Kh10N60K10V10Yu5T3M2B with a diameter of 60 mm

The macrostructure of an unmodified nickel alloy ingot is characterized by large columnar grains (Figure 1a), up to 30 μm in size, in which liquation chemical inhomogeneity and pores are revealed. The modification prevented the formation of columnar grains. The structure is dominated by equiaxed grains up to 1.5 mm in size (Figure 1b).

This may be because titanium carbonitride has high stability in nickel alloys and has a density close to the carbide phase of the alloy.

In the mechanism of structure refinement during this modification, two factors can be distinguished that contribute to this process: mutual blocking and misorientation of dendrites growing in a non-uniform temperature (concentration) field; intensive separation of dendritic branches from the trunk, associated with a non-uniform concentration distribution. The first factor is realized at the growth stage, the second — during the transformation of dendrites when they coexist with the melt in a two-phase state. Introducing a dispersed solid phase into the melt distorts the local temperature and concentration field, which causes a violation of the columnar structure.

Introducing ultrafine particles of titanium carbonitride causes the creation of inclusions in the melt with a concentration and temperature different from the main melt and does not allow the formation of a columnar structure. Changes in the temperature gradient and the rate of movement of the crystallization front significantly affect not only the dendritic structure but also the morphology and topography of the components and the phase composition of heat-resistant alloys.

Conclusions

When modifying nickel alloys, using carbides and nitrides of refractory compounds, primarily titanium as inoculants, is the most rational choice. We achieved the best results when ultrafine particles of titanium carbo nitride were used as inoculators, in an amount of 0.25 wt. % providing a fine-grained structure and a twofold increase in the ductility of castings.

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Е.Н. Еремин

Никельді қорытпаларды түрлендіру үшін, оның құрамын жақсартатын қоспаларды таңдау

Ыстыққа төзімді никель қорытпалары ұшақтар мен өндірістік газ турбиналы қозғалтқыштарға арналған құю өндірісінде кеңінен қолданылады. Құрылымдық факторлар – құйылған никель қорытпаларының пайдалану қасиеттерін анықтаушы болып саналады. Бұл қорытпалардан алынған құймалардың негізгі кемшілігі – ірі түйіршікті құрылым, түйіршіктердің біркелкі еместігі және қимада бағаналы кристалдардың болуы. Сондықтан, қорытпаның оңтайлы құрылымын жасау жоғары қасиеттерді алу және құйылған бөлшектердің сапалы өнімділігін қамтамасыз етудің маңызды шарты болып табылады. Құйылған металдың механикалық және пайдалану қасиеттерінің деңгейіне пайдалы әсер ететін ең әмбебап құрал – бұл ұсақ түйіршікті құрылымды алу. Мұндай құрылымды құрудың ең перспективалы жолы – балқымаға аз мөлшерде кристалл ұйытқысының гетерогенді түзілуін тудыратын қоспаларды енгізу, яғни балқыманы баяу балқитын элементтердің дисперсті бөлшектерімен және инокулярлы–қосылыстарымен түрлендіру. Нақты бір фазаның кристалдануын бастайтын қажетті инокулярлы бөлшектердің түрін таңдау үшін, осындай таңдау принциптері туралы теориялық түсінік қалыптастыруға мүмкіндік беретін мәліметтер жиынтығы болуы керек. Жұмыста құйылған металл құрылымының жасанды өзгеру процесін тудыруы мүмкін инокуляторлар бөлшектерінің түрін таңдау негіздемесі келтірілген. Ыстыққа төзімді никель қорытпасы үшін титан карбонитридінің ультрадисперсті ұнтағының баяу балқитын бөлшектерін инокулятор ретінде қолдану тиімді екендігі көрсетілген. Балқымаға енгізгенде 0,025 салмақ, яғни мұндай бөлшектердің % –ы қорытпаның ұсақтүйіршікті құрылымын алу арқылы қамтамасыз етіледі, ал оның илемділігі түрленбегенмен салыстырғанда екі еседен астам артады.

Кілт сөздер: ыстыққа төзімді никель қорытпасы, балқыма, түрлендіру, кристалдану, инокуляторлар, бөлшектер, қасиеттері, құрылымы.

Е.Н. Еремин

Выбор инокулирующих добавок для модифицирования никелевых сплавов

Жаропрочные никелевые сплавы находят широкое применение в производстве отливок деталей авиационных и промышленных газовых турбин двигателей. Структурные факторы являются определяющими эксплуатационные свойства литых никелевых сплавов. Основным недостатком отливок, полученных из этих сплавов, являются грубокристаллическое строение, разнозернистость и наличие столбчатых кристаллов по сечению. Поэтому создание оптимальной структуры сплава является важным условием получения высоких свойств и обеспечения повышенной работоспособности литых деталей. Наиболее универсальным средством, оказывающим благоприятное влияние на уровень механических и эксплуатационных свойств литого металла, является получение мелкозернистой структуры. Наиболее перспективный путь создания такой структуры — это ввод в расплав небольшого количества добавок, вызывающих гетерогенное образование зародышей кристаллов, т.е. модифицирование расплава дисперсными частицами тугоплавких элементов и соединений-инокуляров. Для выбора типа частиц инокуляров, требуемых для инициирования кристаллизации конкретной фазы, необходимо иметь набор данных, позволяющих сформировать теоретические представления о принципах такого выбора. В работе приведено обоснование выбора типа частиц инокуляторов, способных вызывать процесс искусственного изменения структуры литого металла. Показано, что для жаропрочного никелевого сплава наиболее эффективно использование в качестве инокуляторов тугоплавких частиц ультрадисперсного порошка карбонитрида титана. При введении в расплав 0,025 вес. % таких частиц обеспечивается получение мелкозернистой структуры сплава, а его пластичность, по сравнению с немодифицированным, увеличивается более чем в два раза.

Ключевые слова: жаропрочный никелевый сплав, расплав, модифицирование, кристаллизация, инокуляторы, частицы, свойства, структура.

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