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SURFACE MODIFICATION AND ITS INFLUENCE ON THE MICROSTRUCTURE AND CREEP RESISTANCE OF NICKEL BASED SUPERALLOY RENÉ 77

MODYFIKACJA POWIERZCHNIOWA ORAZ JEJ WPŁYW NA MIKROSTRUKTURĘ I WYTRZYMAŁOŚĆ NA PEŁZANIE ODLEWÓW Z NADSTOPU NIKLU RENÉ 77

Superalloy René 77 is very wide used for turbine blades, turbine disks of aircraft engines which work up to 1050°C. These elements are generally produced by the investment casting method. Turbine blades produced by conventional precision casting methods have coarse and inhomogeneous grain structure. Such a material often does not fulfil basic requirements, which concern mechanical properties for the stuff used in aeronautical engineering. The incorporation of controlled grain size improved mechanical properties. This control of grain size in the casting operation was accomplished by the control of processing parameters such as casting temperature, mould preheating temperature, and the use of grain nucleates in the face of the mould. For nickel and cobalt based superalloys, it was found that cobalt aluminate ($CoAl_2O_4$) has the best nucleating effect. The objective of this work was to determine the influence of the inoculant's content (cobalt aluminate) in the surface layer of the ceramic mould on the microstructure and mechanical properties at high temperature of nickel based superalloy René 77. For this purpose, the ceramic moulds were made with different concentration of cobalt aluminate in the primary slurry was from 0 to 10% mass. in zirconium flour. Stepped and cylindrical samples were casted for microstructure and mechanical examinations. The average grain size of the matrix (γ phase), was determined on the stepped samples. The influence of surface modification on the grain size of up to section thickness was considered. The microstructure investigations with the use of light microscopy and scanning electron microscopy (SEM) enable to examine the influence of the surface modification on the morphology of γ' phase and carbides precipitations. Verification of the influence of $CoAl_2O_4$ on the mechanical properties of castings were investigated on the basis of results obtained form creep tests.

Keywords: Superalloy Renè 77, surface modification, cobalt aluminate, microstructure, creep resistance

Odlewy wykonane z nadstopu René 77 przeznaczone są na elementy części gorącej silników lotniczych (łopatki, tarcze wirnika turbiny). Odlewy tych elementów wytwarzane są najczęściej metodą wytapianych modeli. Łopatki turbin wytwarzane metodami konwencjonalnego odlewania precyzyjnego charakteryzują się gruboziarnistą i niejednorodną mikrostrukturą. Często więc materiał odlewu nie spełnia podstawowych wymagań dotyczących właściwości mechanicznych materiałów stosowanych w technice lotniczej. Poprawę jakości łopatek nadstopów niklu uzyskano poprzez kształtowanie rozmiarów ziarna podczas odlewania. Kontrolę rozmiaru ziarn osiągnięto w wyniku zastosowania odpowiedniej temperatury zalewania, temperatury formy oraz modyfikacji składu chemicznego warstwy wierzchniej formy ceramicznej. Dla nadstopów na osnowie niklu najlepsze efekty rozdrobnienia ziarna uzyskano stosując glinian kobaltu ($CoAl_2O_4$). Celem pracy było określenie wpływu zawartości modyfikatora (glinianu kobaltu) w warstwie wierzchniej formy ceramicznej na mikrostrukturę i właściwości wytrzymałościowe w podwyższonej temperaturze odlewów wykonanych z nadstopu niklu René 77. W tym celu wykonano formy ceramiczne różniące się zawartością glinianu kobaltu w warstwie wierzchniej formy ceramicznej, która wynosiła 0, 5 i 10%mas. Odlano próbki schodkowe do oceny oddziaływania modyfikatora na mikrostrukturę oraz stożkowe do badań wytrzymałościowych. Wyznaczono średnie pole powierzchni płaskiego przekroju ziarna (faza γ) na przekrojach poszczególnych schodków. Uwzględniono również wpływ oddziaływania modyfikatora na rozmiar ziarn w zależności od grubości odlewu (wysokość schodka). Badania mikrostruktury przy użyciu mikroskopu świetlnego i elektronowego skaningowego (SEM) umożliwiły określenie wpływu modyfikacji powierzchniowej na morfologię fazy γ' i węglików. Ocenę wpływu modyfikacji powierzchniowej na właściwości wytrzymałościowe w warstwie wierzchniej odlewu w podwyższonej temperaturze wyznaczono w próbie przyspieszonego pełzania.

1. Introduction

The nickel based superalloy René 77 is used for critical elements of aircraft engines such as turbine blades or turbine discs. These elements are usually produced by the investment casting method, which is used to mass produce parts of turbine engines of a high quality [1, 2]. In the investment casting process an expendable pattern is made, usually by injecting a special wax- based material into a metal mould. Than this pattern is dipped in a ceramic slurry and covered with ceramic

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stucco, left for drying. The dipping and stuccoing are repeated until a mould of necessary thickness is applied. Having completely removing the wax using a steam autoclave the mould is fired. The mould is preheated to a specific temperature and filled with superalloy melt. When a casting is accomplished by the conventional method the shell (mould) is discarded [1, 3]. Turbine blades produced by conventional precision casting method have coarse and inhomogeneous grain structure. Such a material does not fulfil basic requirements, which concern mechanical properties for the stuff used in aeronautical engineering [1, 4]. As to obtain the optimum creep resistance, the incorporation of controlled grain is indispensable. This control of grain size in the casting operation was achieved by the control of process parameters such as casting temperature, mould preheating temperature and by the chemical method of refinement of cast structure. The chemical method of grain refinement of superalloys during casting process is realized by the addition of inoculants (modifiers) into the interior surface of investment mold. Important factors that influence the grain size and the quality of the casting are: the type of modifier and the amount of modifier introduced into the mold. For nickel and cobalt based superalloys it was found that stable phase with spinel structure – cobalt aluminate ($CoAl_2O_4$) has the best nucleating effect. The amount of cobalt aluminate in the primary slurry is highly variable, (ranging from 1 to 10% or higher) and depends on the specification requirements, the alloy being casted, the section thickness, and other factors [5-10].

The goal of the present work was to determine the influence of the inoculant's (cobalt aluminate) content in the surface layer of the ceramic mould on the grain size of the casting made from superalloy René 77. Moreover, the influence of the surface modification on the grain sizes up to its section thickness was taken into consideration.

As mechanical properties of casted elements of nickel based superalloy are determined not only by grains size and orientation but also by morphology and spacing of γ', carbide phases [1, 5] it was examined the influence of surface modification on the morphology of γ' – phase and carbides precipitations. Verification of the influence of $CoAl_2O_4$ on the mechanical properties of castings were investigated on the basis of results obtained from the creep tests.

2. Experimental procedure

The alloy used in this study is René 77. Chemical composition of the used melt is given in Table 1. Stepped and cylindrical samples were cast by precision casting method respectively for microstructure and mechanical properties investigations [10]. In order to achieve the modification effect on the surface layer of the casting the composition of the first layer of ceramic mould undergone changes – reacting directly with the liquid metal. During preparation of ceramic face mould, the suitable content of cobalt aluminate ($CoAl_2O_4$), manufactured by Permedia Lublin company, has been added: 0, 5, 10% mass. in zirconium flour.

TABLE 1
Chemical composition of nickel superalloy René 77 (%mass)

Ni	Cr	Co	Mo	Ta	Al	Ti	C	B	Zr	Fe	S	Si
57	14.61	15.32	4.52	0.05	4.73	3.49	0.07	0.015	0.01	0.08	0.001	0.017

Stepped samples were cut in order to make an observation of microstructure on the cross sections of separate step casting. Polished sections were etched with Marble's reagent ($10gCuSO_4 + 50cm^3HCl + 50cm^3H_2O$). Stereological parameters of microstructure were determined for cross sections of steppes. Macro- examination was performed by the means of the image analysis software APHELION 2.3. The average surface area of grain cross section $\bar{A}$, standard deviation s, and the average shape factor of grains f were calculated. Images of grains of γ matrix were obtained by the means of stereoscope microscope Zeiss Stem 2000-C equipped with the digital camera. Microstructure investigations were performed by the use of light microscope Nikon Epiphot 300 and scanning electron microscope (SEM) HITACHI S-3400N equipped with EDS spectrometer.

Samples of 6.25 mm diameter were machined from cylindrical castings for mechanical properties investigations [10, 11]. Creep tests were made by the use of creep machine W+B Walter+Bai+AG according to ASTM E139 [12] standard. The operating condition of creep tests were as follows: the temperature T= 982°C, and the stress σ_p= 152 MPa. The total time duration of creeping t_z, unit elongation A_4, and percentage reduction of area of the specimen Z were evaluated.

3. Results and discussion

The macrostructure investigations showed that the grain size of γ matrix in the cross section of casting depends on the section thickness of casting and the concentration of $CoAl_2O_4$ in the first layer of ceramic mould (Tab. 2). The average surface area of grain cross section decreases with higher velocity of cooling (the reduction of cross section thickness). For example the average surface area of grain cross section of nonmodified superalloy René 77 on the cross section of casting was 6.38 mm^2 – measured on the cross section with 49 mm of height while for 5 mm of step height it was 1.14 mm^2 (Tab.2). Grain sizes of superalloy reduce with the enlargement of cobalt aluminate concentration in the surface layer of the mould. However, as it is shown in Table 2, the difference is not so significant, as the inoculant content increases above 5%mass. It was found that the average shape factor of grains f remains constant for large sections (of 49 mm). Modification of larger (10%mass.) amount of $CoAl_2O_4$ causes that grains which are formed during crystallization are more elongated in the direction of the heat outflow from the casting. The photos 1a), b) show macrostructure of cross section of the samples of 11 mm thickness. It is visible, that inoculant acts most intensively close to the sample's surface. The macrostructure is not a homogeneous one.

TABLE 2

The stereological parameters of macrostructure of superalloy René 77 depending on the inoculant content and the section thickness (height of a stair)

The section thickness mm	Cobalt aluminate concentration, %mass.								
	0%			5%			10%		
	$\bar{A}$, mm^2	s, mm^2	f	$\bar{A}$, mm^2	s, mm^2	f	$\bar{A}$, mm^2	s, mm^2	f
49	6.38	6.06	2.07	3.79	4.70	1.97	1.49	1.92	2.18
29	6.83	7.52	1.88	4.38	5.79	2.02	2.19	3.32	2.20
23	13.33	11.68	1.71	4.48	5.84	2.00	1.80	3.15	2.57
17	6.62	7.15	2.24	1.50	2.30	2.19	0.86	1.51	2.53
11	3.46	3.90	2.33	1.10	1.49	2.27	0.48	0.56	2.50
5	1.14	1.30	2.10	0.40	0.69	2.18	0.30	0.34	2.78

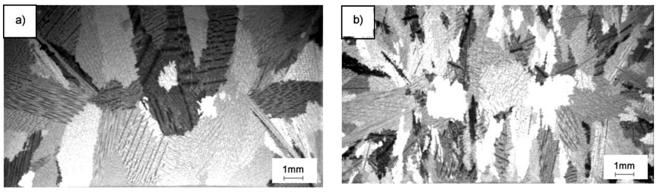

Fig. 1. The macrostructure of the superalloy René 77 on the cross section of casting with 11 mm height: a) nonmodified, b) modified with 5%mass of $CoAl_2O_4$

The microstructure investigations with the use of light microscopy revealed dendritic structure of modified and nonmodified castings, which is typical for that kind of alloys [11]. The carbides precipitations was observed on the boundaries and inside the grains. Fig. 2 presents carbides on grain boundaries. It was established, that modification effects the shape and size of the carbides. In the case of modified microstructure the carbide particles are thinner in comparison to nonmodified ones. The observation of microstructure of superalloy René 77 under the higher magnification with the use of scanning electron microscopy revealed that it consists of super fine – cubic particles of γ' phase (Fig. 3) and austenitic matrix. The influence of the surface inoculation on the morphology and size of γ' phase was not affirmed.

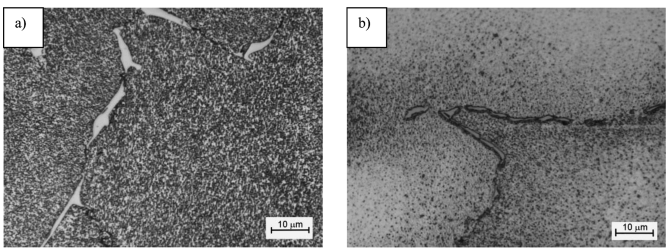

Fig. 2. The microstructure of the superalloy René 77 on the cross section of casting with 49mm height: a) nonmodified, b) modified with 5%mass of $CoAl_2O_4$

Table 3 presents the results of the creep testing. The total time duration of creeping t_z at the temperature 982°C and stress 152 MPa improved. The greater amount of inoculant in the surface layer of ceramic mould did not improve the total time duration of creeping, only plasticity of superalloy slightly droped with 5%mass of $CoAl_2O_4$ and it again rose with 10% mass of cobalt aluminate content.

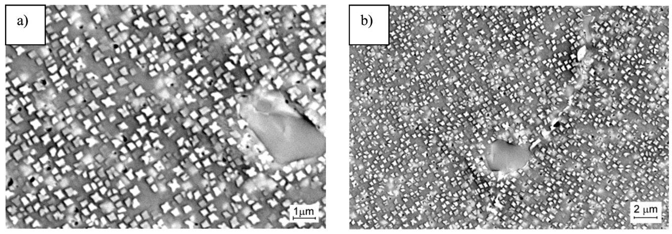

Fig. 3. The microstructure of the superalloy René 77 on the cross section of casting with 11mm height: a) nonmodified, b) modified with 5%mass of $CoAl_2O_4$

TABLE 3

Results of creep test of superalloy René 77

$CoAl_2O_4$ concentration, %mass.	t_z h	A_4 %	Z, %
0	33.3	16	16.24
5	38.5	13.2	15.4
10	38.4	16.65	21

The results of fractures analysis after creep test of René 77 are presented in Fig. 4. One may notice that they are ductile. The occurrence of many dimples with different sizes on their surface means that first small microvoids formed as consequence of plastic deformation and they enlarged to form a crack [13].

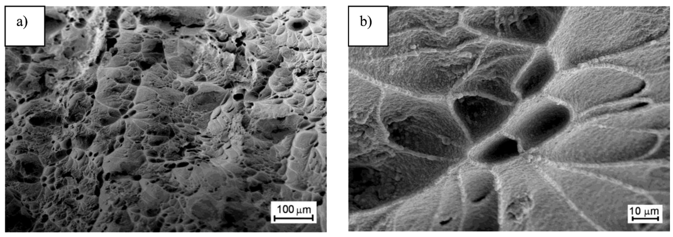

Fig. 4. Fractures of modified superalloy René 77 after the creep tests

The observations of microstructure confirmed that cracking of superalloy at high temperature had intercrystalline character and it occurred along grain boundaries, as shown in

Fig. 5, 6a). During creep, nonmodifed samples had more propensity to carbide coarsening and joining together in grain boundaries in comparison to modified castings, as it is shown in Fig. 5. It could influenced the velocity of crack nucleations and lower creep strength. The growth and directional elongation of γ' phase was observed in all investigated samples (Fig. 6b).

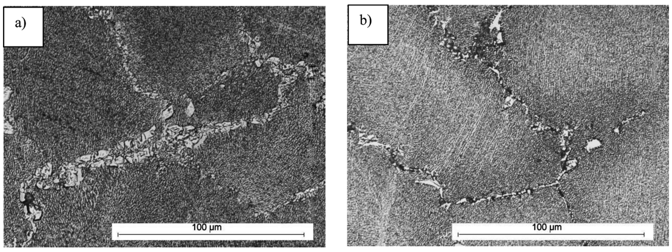

Fig. 5. The microstructure of superalloy René 77 after the creep tests: a) nonmodified b) modified with 5%mass of $CoAl_2O_4$

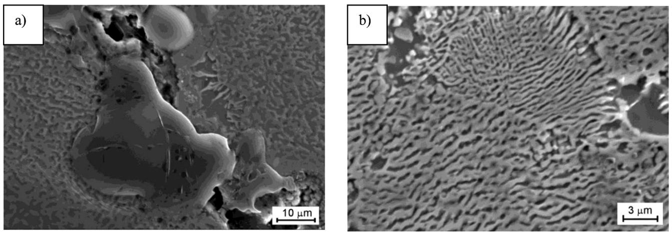

Fig. 6. The microstructure of superalloy René 77 after the creep tests: a) modified, with visible crack along grain boundary b) nonmodified, with visible γ' phase

4. Conclusions

Surface modification of superalloy René 77 results in reduction of grains size of γ matrix and disintegration of carbide precipitates. The inoculant's content has significant impact on the microstructure of superalloy René 77 depending on its section thickness. So the size of γ matrix of the superalloy can be controlled by the inoculant amount added to a slurry.

It was proved, that modification of surface layer of ceramic mould has a positive effect on the mechanical properties at high temperature of the investigated superalloy. Castings with inoculated interior surface of the mould have a higher temperature creep resistances; the rupture time is about 12% longer. The microstructure investigations of the samples after creep tests revealed that mechanical properties of superalloy René 77 are better not only because of the grain refinement but also the carbide refinement.

On the grounds of the obtained results it was found that the optimal concentration of cobalt aluminate powder in ceramic mould to produce casting elements made from superalloy René 77 is about 5-6%mass. The higher concentration of modifier does not change the grain size significantly and does not improve mechanical properties of castings.

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