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Ceramic filters for bulk inoculation of nickel alloy castings

F. Binczyk*, J. Śleziona, P. Gradoń

Chair of Materials Technology Silesian University of Technology, Krasińskiego Str. 8, 40-019 Katowice, Poland * Corresponding author. E-mail address: franciszek.binczyk@polsl.pl

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Abstract

The work includes the results of research on production technology of ceramic filters which, besides the traditional filtering function, play also the role of an inoculant modifying the macrostructure of cast nickel alloys. To play this additional role, filters should demonstrate sufficient compression strength and ensure proper flow rate of liquid alloy. The role of an inoculant is played by cobalt aluminate introduced to the composition of external coating in an amount from 5 to 10 wt.% . The required compression strength (over 1MPa) is provided by the supporting layers, deposited on the preform, which is a polyurethane foam. Based on a two-level fractional experiment 2^{4-1} , the significance of an impact of various technological parameters (independent variables) on selected functional parameters of the ready filters was determined. Important effect of the number of the supporting layers and sintering temperature of filters after evaporation of polyurethane foam was stated.

Keywords: Nickel superalloys, Ceramic filter, Inoculation, CoAl₂O₄ inoculant, Compression strength, Density, Porosity

1. Introduction

Currently, precision castings for parts of aircraft engines are made from modern grades of nickel and cobalt alloys such as IN 100 and IN 713C, RENE 77, MAR-M257 and MAR M 509 [1, 2]. From castings made of these alloys, tight dimensional tolerances, excellent surface quality in as-cast state and after heat treatment as well as minimum gas porosity and shrinkage effects are expected. Efforts are also made to obtain the structure of equiaxial grains within the entire volume of the casting

World literature gives a lot of information on microstructure improvement in nickel superalloys by refining and inoculation with nanoparticle inoculants [3-5].

The main parameters determining the properties and thus the quality of nickel alloy castings are metallurgical quality and proper structure of the casting. The alloy purity can be achieved through the use of appropriate ceramic filters. In this way, considerable fraction of the oxide impurities are arrested in the internal space of the filter. In most cases, these inclusions have their origin in the poor quality of charge materials, in improper lining of the induction furnace and oxidation during melting in the case of low vacuum or improper protective atmosphere. The reference literature, both national and international, gives a lot of important information on the effectiveness of ceramic filters and the quality of castings obtained [6-10].

If the surface of the filter contains an inoculating component, its presence should influence the structure of the casting by bulk inoculation, which will produce in this casting the structure of fine equiaxial grains, impossible to obtain by standard surface inoculation, after which some columnar grains always remain in the casting interior, deteriorating considerably the mechanical properties.

To make the filter capable of playing this role, it should possess sufficient mechanical and thermal resistance. World literature gives a variety of information on the use of ceramic filters. For economic reasons, the cost of using a filter has to be compensated with the benefits of clean metal and correct structure, obtained owing to the use of this filter. Therefore it is important to develop technologies that will enable manufacture of high-strength filters, capable of performing at the same time the role of filtering and inoculating-refining units [11]. Studies towards the development of such filters have been taken under the key project POIG.0101.02-00-015/08 in the Operational Programme Innovative Economy (OPIE).

2. Plan of research

The aim of the study was to evaluate the significance of the effect of selected technological parameters (independent variables) on the functional properties of inoculating filters.

The study was based on a fractional experiment 2^{4-1} , requiring eight experiments. The plan is presented in Table 1.

Table 1.

Plan of fractional experiment 24-1

Experiment	Independent variable					
2.1.p.01.1.0.1.	А	В	С	D		
1	10	2	150	600		
2	16	2	150	800		
3	10	4	150	800		
4	16	4	150	600		
5	10	2	300	800		
6	16	2	300	600		
7	10	4	300	600		
8	16	4	300	800		

The following parameters were adopted as independent variables :

A: The number of supporting layers (REMASOL + zirconium flour),

B: The number of inoculating layers (colloidal silica + zirconium flour + cobalt aluminate),

C. Foam evaporation temperature,

D. Sintering temperature.

The following parameters were adopted as dependent variables : • compression strength, MPa ,

- porosity,%
- loose material flow rate through filter, cm^3 / s ,

3. Making filters

Stage I:

The first step is to prepare an appropriate mixture of binder ("Remasol") and zirconia powder. Then, the successive supporting layers are applied in the following sequence:

• pouring the ready ceramic slurry through preform (polyurethane foam),

- blowing the preform with compressed air (to protect the filtrating holes from getting stuck),
- drying.

These operations are repeated the number of times corresponding to the number of supporting layers. According to the prepared plan of experiments, 10 or 16 supporting layers were prepared. This step is illustrated in Figure 1.



Fig. 1. Preform before and after the application of supporting layers

Stage II:

After drying, onto the last supporting layer, a layer of inoculant is applied. It is composed of a mixture of colloidal silica, powdered zirconium and cobalt aluminate, all mixed in appropriate ratios. The method of applying the inoculating layers is the same as for the supporting layers, namely:

- pouring the ready ceramic slurry through preform.
- blowing of preform with compressed air,
- drying

A filter after the application of inoculating layers is shown in Fig. 2.



Fig. 2. Filter after the application of inoculating layers

Stage III:

The last step in the manufacture of inoculating ceramic filters is evaporation of preforms and sintering of the supporting and inoculating layers. The evaporation of polyurethane foam was conducted at 150°C and 300°C, while sintering took place at 600°C and 800°C. The processes were carried out in a resistance electric oven. In each case, samples were placed in the oven for 30 minutes. Cooling took place together with the oven. Examples of the ready filters are shown in Figure 3



Fig. 3. The ready inoculating ceramic filters

4. Measurement of filter properties

The following properties of filters were examined: Rc strength, density, porosity and loose material flow rate. The results of these measurements are shown in Figures 4 to 7.



Fig. 4. Compression strength compared for different filters



Fig. 5. Porosity compared for different filters



Fig. 6. Comparison of loose material flow rate through filter

5. The results of investigations and discussion of results

The effect of dependent variables (variables A, B, C and D) on selected properties of the filters was evaluated by multiple regression analysis. The level of significance $\alpha = 0.1$, i.e. the probability of committing an error of first kind at a level of 0.1, was adopted. How strong the impact of the examined technological parameter will be depends on the value of probability p, while the direction of influence (decrease or increase) depends on the sign preceding the coefficient b_0 (- or +). Calculations were carried out using a licensed StatSoft V.7.1 Pl. Statistica software. Statistical calculations were performed on actual values of the independent variables.

A regression summary for the dependent variables: compression strength $R_{c,}$, porosity and flow rate is shown in Table 2.

Table 2.

Reg	ression	summary :	for t	he c	lepen	den	t v	ariabl	es
					n	1.			

	Resultant property					
Parameter	Rc, MPa		Porosity, %		Intensity, cm ³ /s	
	bo	р	bo	р	bo	р
Absolute term B	-1,263	0,008	97,78	0,001	4,225	0,001
А	0,098	0,001	-1,68	0,002	-0,16	0,001
В	0,024	0,341	-0,53	0,335	0,018	0,626
С	-0,001	0,062	0,01	0,258	0,001	0,128
D	0,001	0,012	-0,002	0,685	0,000	0,422
\mathbb{R}^2	0,9704		0,9438		0,9675	

Based on the evaluated impact of various technological parameters on compression strength, the following has been stated.

The number of supporting and inoculating layers and the sintering temperature increase the compression strength (positive value of coefficient B), while higher evaporation temperature reduces this parameter (negative value of coefficient B). As the probability value p indicates, a statistically significant effect on Rc exert in descending order the following parameters: the number o f supporting layers, sintering temperature and

evaporation temperature. Statistically insignificant is the number of inoculating layers.

The value of the adjusted coefficient of determination R 2 = 0.9704 indicates that approximately 97% of the results can be explained with a model described by the following relationship:

$R_c = -1,263 + 0,098 \cdot A - 0,0008 \cdot C + 0,0011 \cdot C$

Based on the evaluated impact of various technological parameters on porosity, the following has been stated. The porosity of filters is significantly affected only by the number of supporting layers in descending order of their occurrence (negative value of coefficient B). Other parameters have not a significant influence ($p > \alpha$).

The value of the adjusted coefficient of determination R 2 = 0.9438 indicates that about 94% of the results can be explained with a model described by the following relationship:

porosity = 97,78 - 1,68 · A

Based on the evaluated impact of various technological parameters on the sand flow rate through filter, the following has been stated.

The sand flow rate through filter is significantly affected only by the number of supporting layers in descending order of their occurrence (negative value of coefficient B). Other parameters have not a significant influence $(p > \alpha)$.

The value of the adjusted coefficient of determination R 2 = 0.9675 indicates that approximately 97% of the results can be explained with a model described by the following relationship:

Sand flow rate through filter $= 4,225 - 0,155 \cdot A$

6. Conclusions

- 1. Based on the conducted regression analysis, a significant effect of the number of supporting layers and of the sintering temperature on the functional properties of filters was stated.
- 2. The effect of the number of inoculating layers and polyurethane foam evaporation temperature is less significant.
- 3. The required compression strength (above 1 MPa) for ceramic filters with an inoculating coating is obtained after application of at least 16 supporting layers.
- 4. The most advantageous properties are provided by the supporting layers based on zirconium silicate bonded with "Remasol" binder.
- 5. The gasification temperature of polyurethane foam should not be higher than 200°C, while the filters baking temperature should be from 800 to 1000°C.
- 6. It is recommended to apply 2 to 3 inoculating layers containing zirconium silicate, from 7 to 10% cobalt

aluminate, and from 2 to 4% Al powder. A small addition of hafnium powder is also recommended. As a binder for these components it is recommended to use colloidal silica.

7. Thus made filters provide the effect of bulk inoculation, eliminating to a significant degree the incomplete effect of surface inoculation.

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