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Effect of modification and cooling rate on the microstructure of IN-713C alloy

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Abstract

The results of investigations of the effect of modification and cooling rate on the microstructure of castings made from IN-713C nickel superalloy were described. As an inoculant, cobalt aluminate $CoAl_2O_4$ in composition with aluminium powder and colloidal silica was used. Changes in the cooling rate were obtained by the use of cast stepped test piece with steps of 6, 11 and 17 mm thickness. The phase and chemical composition of microstructural constituents, i.e. of γ phase, γ' phase and eutectic carbide precipitates, was evaluated. A significant effect of the cooling rate and modification treatment on the stereological parameters of carbide precipitates was confirmed. Problems in evaluation of the chemical composition of these precipitates in the case of a high degree of the structure refinement were indicated.

Keywords: Nickel superalloys, Microstructure, Cooling rate, Modification, Microanalysis

1. Introduction

The structural „hot parts" of aircraft engines are subject to quite exceptional requirements regarding the manufacturing process regime and quality. At present, the near-net-shape castings of the aircraft engine parts are made from modern grades of nickel and cobalt alloys, like RENE-77, IN-100, IN-713C [1, 2]. On solidification, these alloys produce a specific type of macrostructure, composed of frozen and columnar grains. Structure of this type is prone to crack formation and propagation, resulting in fatal failure of the aircraft engines [3, 4]. Therefore, every attempt should be made to obtain the structure of equiaxial grains within the whole casting volume.

The technical world literature provides comprehensive information on how to refine the microstructure of nickel superalloys. A simple solution is surface modification with inoculant placed in a layer directly reproducing the casting surface [5. The authors of the present study were investigating the solidification process [6] and the effect of volume modification of an IN-713C nickel superalloy on the stereological parameters of its macrostructure [7-9]. The results presented in these studies can serve as a good example of the beneficial effect that an inoculant composed of cobalt aluminate, aluminium powder and colloidal silica is expected to have on the crystallisation and refinement of equiaxial grains. Considering the lack of more detailed data on the effect that volume modification and casting cooling rate are supposed to exert on alloy microstructure, studies were undertaken on this particular subject, with attention focussed on the X-ray microanalysis of microstructural constituents.

2. Materials and methods of investigations

Studies were conducted on an IN-713C nickel superalloy which, besides nickel, also contained: 0,03% Co, 13,26% Cr, 5,85% Al, 4,10% Mo, 0,85% Ti, 2,27% (Nb + Ta) and 0,12%C.

Melts were made in an Al_20_3 crucible of Balzers VSG-02 induction furnace. Stepped test pieces were cast to evaluate the effect of cooling rate on macro- and microstructure of the individual test piece steps. The casting designed by WSK Rzeszów had the dimensions adjusted to the size of a vacuum furnace chamber. Moulds, before being put in furnace chamber, were preheated to 750^oC. The temperature of molten metal and of ceramic mould was controlled with a Pt-PtRh10 immersion thermocouple. The alloy pouring temperature ranged from 1400 to 1500^oC.
The inoculants were prepared from cobalt aluminate $CoAl_2O_4$, aluminium powder, and zircon flour, all mixed in different proportions. As a binder, the colloidal silica was used. The product was crushed after drying. Several experiments were made, varying the temperature of pouring and inoculant location, and achieving due to this different molten alloy/inoculant contact times.
As a criterion in evaluation of the cooling rate and modification effect on microstructure formation, the results of an X-ray microanalysis of the main phase constituents were used. Specimens were taken from the "steps" of the cast stepped test piece of 6, 11 and 17 mm thickness and were used in the following experimental variants:
1. Alloy without modification, pouring temperature 1480^oC.
2. Inoculant in an amount of 1g placed on filter, pouring temperature 1500^oC.
3. Inoculant in an amount of 1g placed on aluminium foil between filters, pouring temperature 1440^oC.
4. Inoculant in an amount of 1g placed between filters, pouring temperature 1420^oC.
5. Inoculant in an amount of 0.5 g placed between filters (with side hole), pouring temperature 1400^oC.
To identify the macro- and microstructure, the specimens were etched with a reagent of the following composition: 18 g/l HNO_3, 280-320 g/l HCl, 151-173 g/l $FeCl_3$).

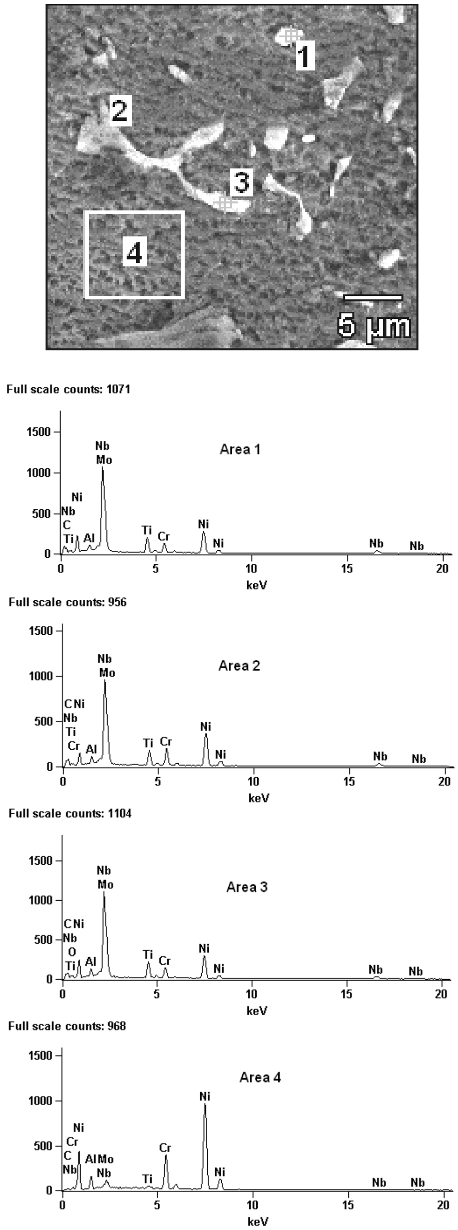
3. The results of investigations and discussion

The results of the investigations were discussed taking as an example castings from melts nos. 1 and 4. The qualitative and quantitative microanalyses were carried out on an EDS X-ray spectrometer made by Thermo Noran, equipped with Noran System SIX for microanalysis, cooperating with a Hitachi S-3400N scanning microscope.

For each of the examined specimens, the microstructural images were prepared, a qualitative analysis of the chemical composition in selected regions was performed, together with a quantitative analysis of the chemical composition of the individual microstructural phase constituents, i.e. of γ phase, γ' phase, and carbides.

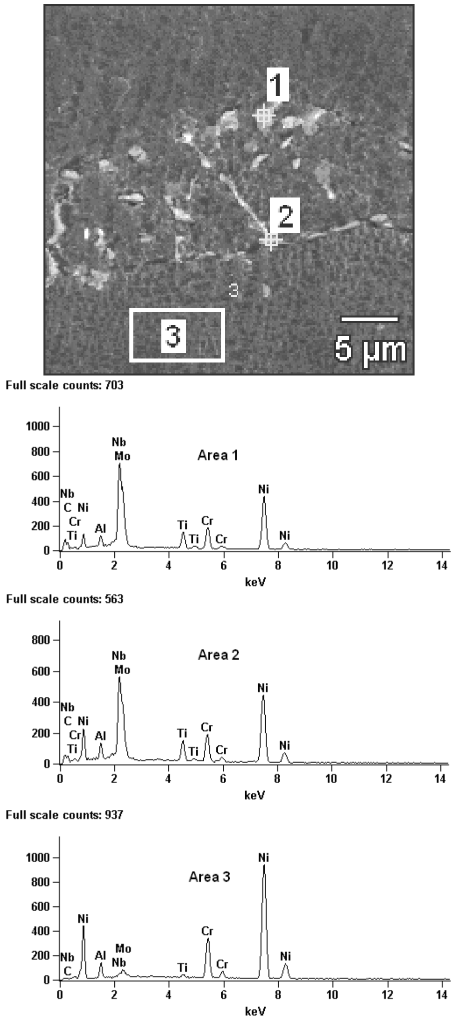
The results of such analyses for specimens taken from the step of 17 mm thickness are shown in Figs. 1 and 2 for melts nos. 1 and 4, respectively.

Basing on the results of an X-ray microanalysis carried out in selected microregions (carbides and matrix), the respective graphs were plotted to compare the content of individual constituents present in the examined IN-713 C alloy. The results of this comparison are shown in Figs. 3 to 7.



Area	Al	Ti	Cr	Ni	Nb	Mo
1	1,99	7,77	5,84	21,07	53,90	9,44
2	1,86	5,94	7,79	28,93	47,06	8,41
3	2,23	7,44	6,31	21,98	51,53	10,50
4	6,21	0,66	14,63	71,80	2,20	4,50

Fig. 1. The results of qualitative and quantitative microanalyses in selected regions of 17 mm sample from melt no. 1



Area	Al	Ti	Cr	Ni	Nb	Mo
1	2,86	5,60	8,11	33,00	38,17	12,25
2	3,30	5,20	8,71	39,27	32,87	10,65
3	5,75	0,97	13,30	75,23	1,59	3,16

Fig. 2. The results of qualitative and quantitative microanalyses in selected regions of 17 mm sample from melt no. 4

In 6 mm samples, the process of crystallisation was proceeding much more rapidly than in samples of larger dimensions (11 and 17 mm). This is why the macrostructure of such samples has revealed a higher content of the very fine equiaxial grains. This, in turn, results in higher hardness and probably better combination of mechanical and useful properties.

It has been noticed that the results of the chemical analysis of the precipitates of eutectic carbides present in the 6 mm thick step of a cast stepped test piece are less reliable than the results obtained on samples of larger dimensions, i.e. 11 and 17 mm. As follows from Figs. 3 to 5, elements like Ni, Cr and Al are present, first of all, in the metallic matrix, which is composed of γ phase and an ordered γ' phase $(Ni, Cr)_3Al$.

In the 6 mm thick samples, the analysis of the eutectic carbide precipitates is very difficult, because of a high degree of the structure refinement. In cases like this, the electron beam is "sweeping" not only the regions where carbides are present but also fractions of the metallic matrix. This is why the high content of Ni and Cr has been detected in carbide precipitates. With increasing overall dimensions of the sample, the dimensions of the carbides are also growing, and the results of the analysis are coming closer to the reality. Lower content levels of Ni, Al and Cr were detected. Depending on the dimensions of samples taken from different melts, different types of microstructure were observed.

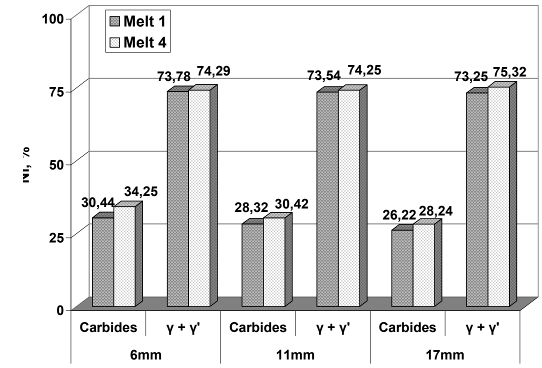


Fig. 3. Comparison of Ni content in carbides and matrix

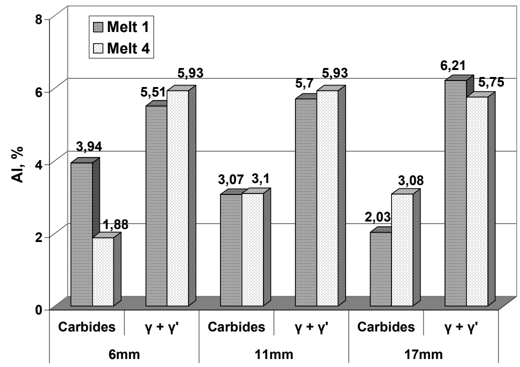


Fig. 4. Comparison of Al content in carbides and matrix

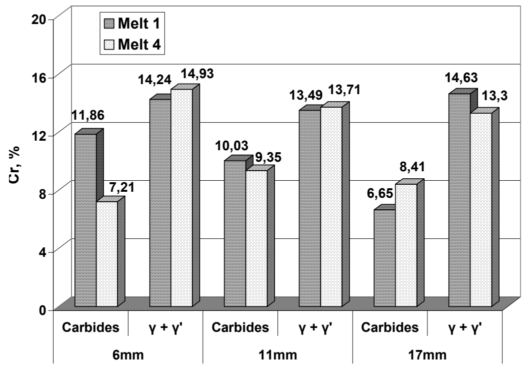


Fig. 5. Comparison of Cr content in carbides and matrix

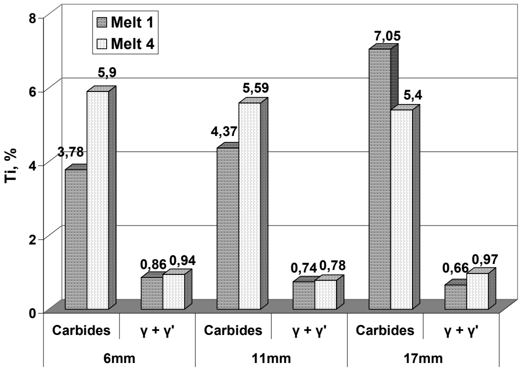


Fig. 6. Comparison of Ti content in carbides and matrix

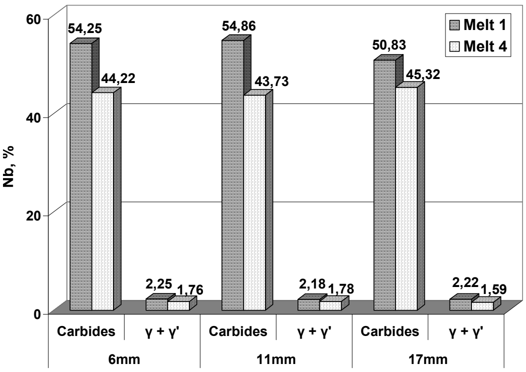


Fig. 7. Comparison of Nb content in carbides and matrix

Under such circumstances, the chemical analysis of carbides can be performed more successfully than in the case of 6 mm samples. The presence in carbide precipitates of elements like Nb, Ti and Mo indicates that these are the primary carbides of MC type (MoC, TiC) and eutectic $M_{23}C_6$ carbides, containing mainly Nb, Mo and Ti.

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