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Macro- and microhardness of IN-713C nickel superalloy constituents

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Abstract

The results of investigations of the effect of modification and cooling rate on the macrohardness of castings and microhardness of phase constituents in 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 using a cast stepped test piece with steps of 6, 11 and 17 mm thickness. Macrohardness of the cast test piece steps was measured by Brinell technique, while Vickers method was used to measure the microhardness of γ and γ' phases present in the alloy matrix, as well as the hardness of eutectic carbide precipitates. A significant effect of the cooling rate and modification treatment on the results of the measurements was stated, and difficulties in performing correctly the microhardness measurements due to the precipitates dimensions, especially after the modification treatment, were highlighted.

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

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. One of the solutions is surface modification with inoculant placed in a layer directly reproducing the casting surface [5]. Unfortunately, surface modification does not satisfy the designers' expectations. It has been proved that the macrostructure of castings poured from an IN-713C nickel alloy in moulds with the inoculating coating undergoes modification either on the casting surface alone or, if this effect happens to penetrate inside, it is to a very small depth only. In central regions of the casting, the undesired structure of columnar crystals remains intact [7-10].

The authors of the present study were investigating the solidification process [7] and the effect of volume modification of an IN-713C nickel superalloy on the stereological parameters of its macrostructure [8, 9]. The results presented in these studies can serve as a good example of the beneficial effect that volume modification of an alloy is expected to have on the crystallisation and refinement of equiaxial grains. Good results were obtained from modification with a mixture composed of cobalt aluminate, aluminium powder and colloidal silica, used as a binder. The authors indicated the modifying effect of cobalt particles [10].

In this study an attempt has been made to determine the combined effect of volume modification treatment and cooling rate on macrohardness of castings and microhardness of the phase constituents in an IN-713C nickel alloy. For this purpose, the authors used a cast stepped test piece with elements of varied thickness.

2. Material 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°C. 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°C. Figure 1 shows a general view of the test stand.

Fig. 1. A view of the vacuum induction furnace, model VSG-02

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°C.
2. Inoculant in an amount of 1g placed on filter, pouring temperature 1500°C.
3. Inoculant in an amount of 1g placed on aluminium foil between filters, pouring temperature 1440°C.
4. Inoculant in an amount of 1g placed between filters, pouring temperature 1420°C.
5. Inoculant in an amount of 0.5 g placed between filters (with side hole), pouring temperature 1400°C.

Figure 2 shows a schematic representation of the main technological variants of alloy modification.

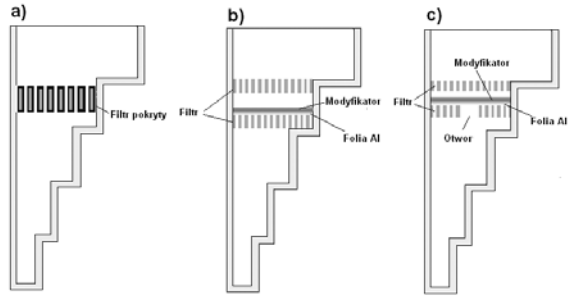

Fig. 2. Schematic representation of the modification variants: a) inoculant on ceramic filter, b) and c) inoculant on foil between filters

For microstructure identification, 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

From the 6, 11 and 17 mm thickness „steps" of the cast stepped test piece, specimens were taken for polished metallographic sections and for the measurement of macro- and microhardness of the individual phase constituents present in alloy matrix. A comparison of macrostructures in specimens before and after modification is shown in Fig. 3.

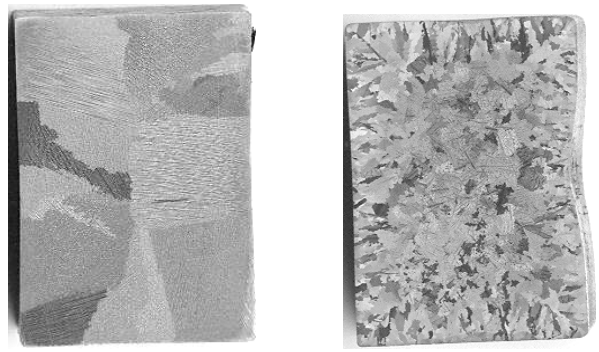

Fig. 3. Comparison of macrostructures in 17 mm thick specimens from experiments 1 and 4

Macrohardness was measured with Rockwell-Brinell hardness tester, type KP 15002P. The hardness tester operating parameters were as follows:

- indenter diameter – 2,5 mm,
- force pressure – 1875 N,
- measurement time – 12 seconds.

For each examined specimen, five measurements were taken; the calculated mean values are shown in Figure 4.

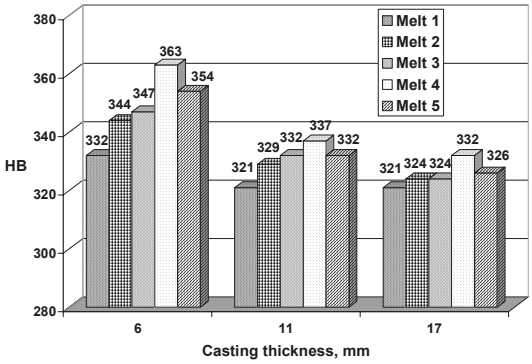

Fig. 4. Comparison of Brinell hardness in the examined specimens

An example of microstructure in 17 mm thick specimen of the stepped test piece cast from melt no. 4 is shown in Figure 5.

a)

b)

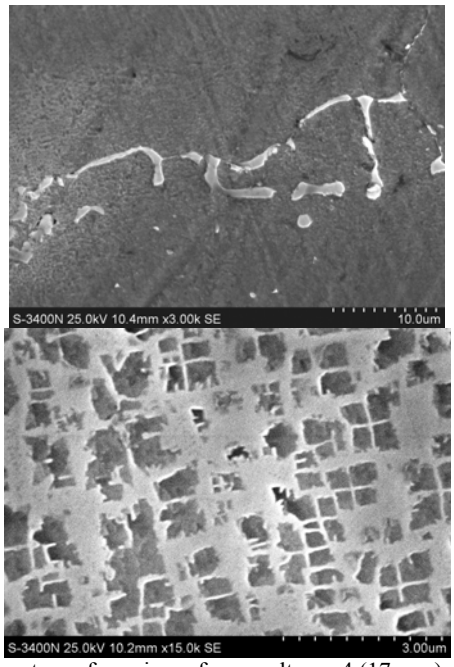

Fig. 5. Microstructure of specimen from melt no. 4 (17 mm): a) matrix + carbides, b) matrix (γ + γ' phase)

The microhardness of phase constituents was measured with Struers Duramin-2 hardness tester. The hardness tester operating parameters were as follows:

- component lens magnification – 40x,
- eyepiece magnification – 10x,
- force pressure – 0,25 N,
- measurement time – 5 seconds.

Figures 6 to 10 show mean results of the five measurements taken for individual microstructural phase constituents, i.e. γ phase, γ' phase, and carbides, on specimens from the successive melts.

The aim of the conducted studies was to investigate the effect of cooling rate and modification on hardness of castings and microhardness of the alloying constituents in IN-713C alloy. The obtained results indicate that increased cooling rate raises the macrohardness of samples, in both modified and unmodified condition, its effect on unmodified samples being, however, much less prominent, compared to the modified ones.

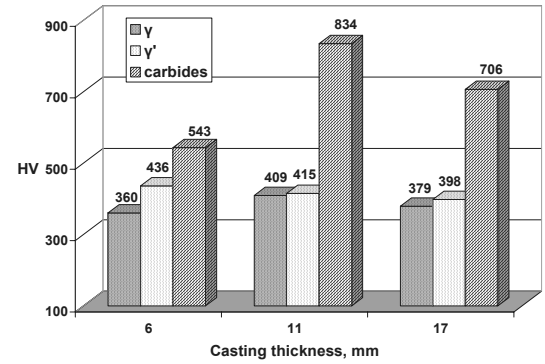

Fig. 6. Comparison of hardness values in the microstructural phase constituents of casting from melt no. 1

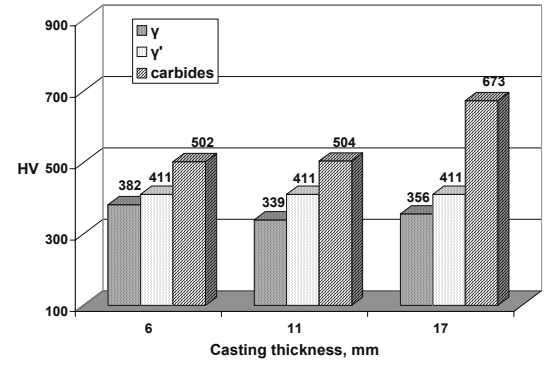

Fig. 7. Comparison of hardness values in the microstructural phase constituents of casting from melt no. 2

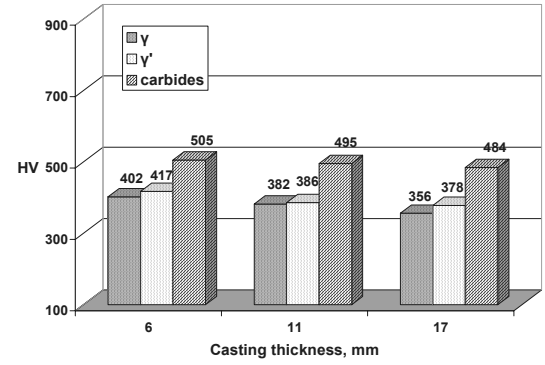

Fig. 8. Comparison of hardness values in the microstructural phase constituents of casting from melt no. 3

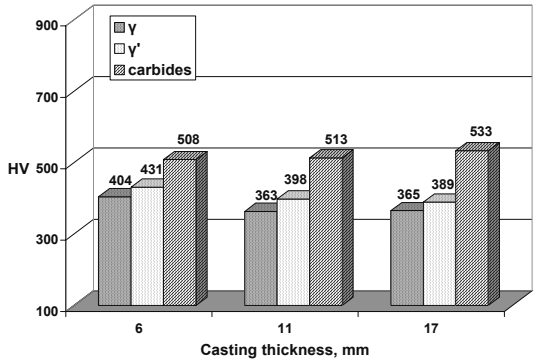

Fig. 9. Comparison of hardness values in the microstructural phase constituents of casting from melt no. 4

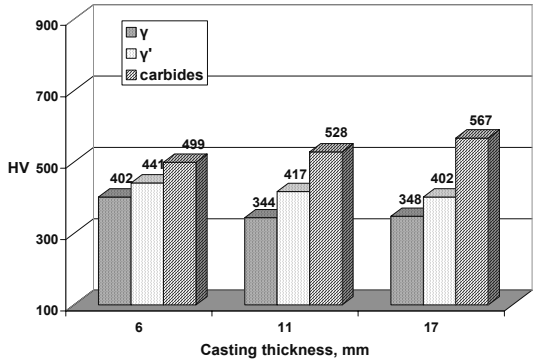

Fig. 10. Comparison of hardness values in the microstructural phase constituents of casting from melt no. 5

This effect is more visible in the results of the macrohardness measurements. In measurements of the alloying constituents microhardness, some discrepancies have occurred in the values obtained for 11 mm and 17 mm thick specimens.

The results of the measurements indicate that longer time of the high rate cooling eliminates the temperature effect on hardness, as can be seen in 17 mm thick samples characterised by similar Brinell hardness values.

Macrohardness of specimens decreases with increasing temperature of pouring. On the other hand, the temperature of pouring depends on the quantity and location of the inoculant, and therefore it is not possible to clearly state if it is the temperature of pouring that is most important or the quantity, possibly also the location, of an inoculant. The analysis of the microhardness of the alloying constituents yielded the expected results. The phase of the lowest hardness was γ phase, followed by γ' phase. The highest hardness had the eutectic carbides, but when it was measured, some problems of strictly technical nature have occurred. The indenter of a Struers Duramin-2 hardness tester at a maximum obtainable magnification of 400 times was penetrating more into the γ phase, leaving the chain-like carbide precipitates mostly untouched. The high values of the carbide hardness could be obtained only when the indenter was deliberately placed in a cluster of the single carbide chains.

Acknowledgements

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