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

F. Binczyk[1]*, J. Śleziona[1], A. Kościelna[2]
[1]Chair of Metal Alloys and Composites Engineering, [2]Chair of Materials Science
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 results of investigations of the effect of modification and cooling rate on the macrostructure of castings made from IN-713C nickel superalloy were described. As a modifier, 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 a stepped test piece with the steps of 6, 11, 17 and 23 mm thickness. As a criterion for the evaluation of casting macrostructure, the stereological parameters, like grain count, relative surface area, shape factor, and indeces of the grain size and shape homogeneity, were applied. The modification treatment was observed to change the grain type from columnar to equiaxial. The stereological parameters of the equiaxial grains depended to a great extent on the cooling rate of the individual elements of a cast stepped test piece.

Keywords: Nickel superalloys, Macrostructure, Cooling rate, Modification, Equiaxial grains, Shape factor

1. Introduction

The structural „hot parts" of an aircraft engine are subject to quite exceptional requirements as regards the manufacturing process and quality control regime. 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. A 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 a comprehesive information on how to refine the microstructure of nickel superalloys [5-11]. A simple solution is surface modification with inoculant placed in a layer directly reproducing the casting surface [5}

In [6] the results of modification of an Inconel 718 alloy with microadditions of cobalt oxide CoO were presented. A minor degree of structure refinement with slight improvement of the mechanical properties was obtained. In [7] the authors were discussing the results of the investigations relating to a modification process of nickel superalloys with inoculants containing tungsten oxides and carbides. The results provided by [8] can serve as an example of the favourable effect of boron microadditions (introduced in an amount of up to 0,01%) on the refinement of microstructure and further extension of the region of the equiaxial crystals.

Recently, numerous publications have appeared on the subject of refining and modification of nickel superalloys with complex systems of intermetallic phases formed of elements included in the group of Fe, Cr, Co and Nb [9-11]. Particularly favourable results were obtained for a mixture of the intermetallic phases of Co_3FeNb_2 and Cr FeNb [11].

Considering the lack of more detailed data on the effect that volume modification is supposed to have on the macro- and microstructure of castings made from nickel superalloys, within a commissioned research project, studies were undertaken on this particular subject. Data are also lacking on the effect that modification is supposed to have on the morphological features of the nickel superalloys macrostructure.

2. The research problem

Surface modification of casting (with inoculant placed in the face layer of ceramic mould) cannot satisfy the designer's expectations; moreover, the mechanism by which this treatment is affecting the primary structure of castings made from the above mentioned alloys is practically unknown [5].

Examples of macrostructures obtained in the individual steps of a test piece (IN-713C alloy) cast in a ceramic mould based on zircon flour and coated inside with an inoculant (zircon flour + 10% cobalt aluminate) are shown in Figure 1.

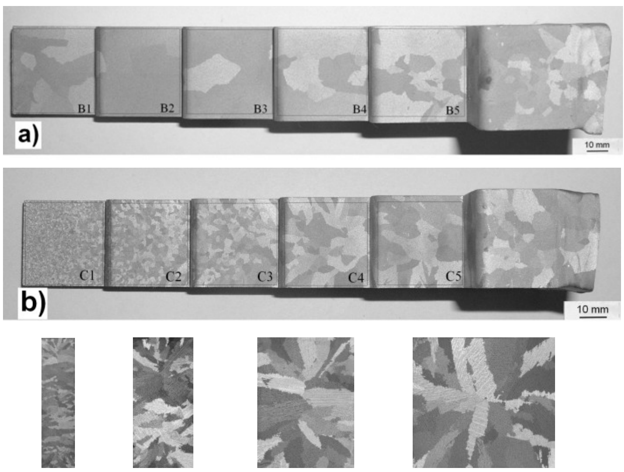

Fig. 1. Macrostructure on the surface and cross-section of a cast stepped test piece: a) alloy without modification, b) alloy after surface modification

The individual elements of a stepped test piece cast in mould without the inoculant coating have the grains definitely coarser than the grains produced in casting poured in the mould with inoculant coating. Moreover, in non-modified castings, the cooling rate has no effect on the grain size. Macrostructures on the cross-sections show a very superficial modification effect, in the best case penetrating inside the casting to a very small depth only. In direction towards the casting centre, the presence of the highly undesired columnar crystals is noticed. So far, the modifying effect of the inoculants ($CoAl_2O_4$) has not been fully explained.

In [12-14] it has been observed that when the liquid metal is poured into a mould coated inside with inoculant, there is a reaction of exchange taking place between $CoAl_2O_4$ and active elements present in the examined superalloys (among others, also Al):

$$CoAl_2O_4 + 2/3Al = Co + 4/3\ Al_2O_3$$

As long as they remain undissolved in liquid alloy, the particles of cobalt (groups or clusters) may act as nuclei. Therefore, the lower is the temperature and the shorter is the time of contact, the less of the particles will dissolve in liquid alloy until the moment when its solidification starts. This is the reason why in thin-walled castings, cooling more rapidly (with higher undercooling), the number of the surface nuclei that remain undissolved in liquid metal is larger, and due to this, the effect of surface modification is stronger.

It seems advisable to investigate more thoroughly the process of volume modification, producing the nuclei of crystallisation still before the alloy enters mould cavity, e.g. in the gating system when the alloy is passing through a filter.

3. Materials and methods of investigation

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. Melting was carried out in an $Al_2 0_3$ crucible of Balzers VSG-02 induction furnace. Stepped test pieces of dimensions adjusted to the size of the induction furnace chamber were cast. Due to this, it was possible to evaluate additionally the cooling rate effect on the grain size. Before being placed in the furnace chamber, moulds were preheated to 750^{o}C. The liquid metal temperature and the ceramic mould temperature were controlled with a Pt-PtRh10 immersion thermocouple. The alloy pouring temperature was from 1400 to 1500^{o}C.

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 changing in this way also the molten alloy/inoculant contact time. The criterion used for evaluation of the modification effect was macrostructure obtained in the stepped test pieces of 6, 11 and 17 mm thickness. In the study, the results of the following experiments were presented:

1. Alloy without modification, pouring temperature - 1480^{o}C.
2. A 1 gram inoculant sample placed on filter, pouring temperature - 1500^{o}C.
3. A 1 gram inoculant sample placed on aluminium foil between filters, pouring temperature - 1440^{o}C.
4. A 1 gram inoculant sample placed between filters, pouring temperature – 1420^{o}C.
5. A 0.5 gram inoculant sample placed between filters (with side hole), pouring temperature - 1400^{o}C.

4. The results of investigations and discussion of results

Macrostructural examinations (specimens etched with Marble's reagent) were carried out under a Nikon Epiphot 200 optical microscope. Figure 2 shows examples of the results obtained on specimens taken from the casting of 17 mm wall cross-section (experiments 1 and 4). The computer analysis of binary images, enabling the grain size and shape to be evaluated, was made by MetIlo image analysis program [15]. The following parameters were used in description of the grain size and shape:

- average plane grain section surface area $\bar{A}$, mm^2,
- average grain perimeter, mm
- specific grain surface area S_v, mm^{-1},
- dimensionless shape factor $\xi = 4\pi A/P^2$

  where: A – plane grain section surface area,
  P - plane grain section perimeter.

The results of computations of the main stereological parameters of the grains in cast macrostructure are shown in Figs. 3 to 6. Figure 2 and respective images of the IN-713C alloy cast macrostructure definitely confirm the beneficial effect that the in-mould volume modification process has on the elimination of columnar grains and crystallisation of the equiaxial ones.

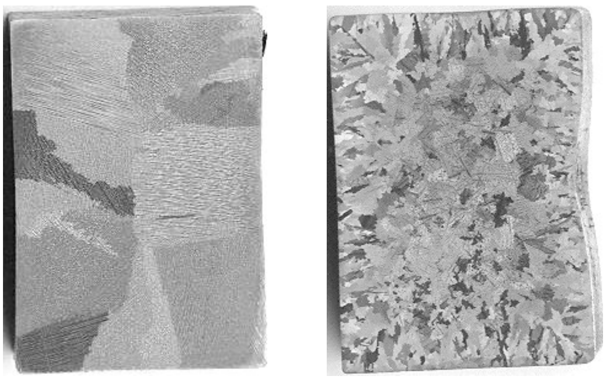

Fig. 2. Comparison of macrostructures in 17 mm thick specimens from experiments 1 and 4

Volume modification always refines the alloy macrostructure, but intensity of this effect depends on the way in which the treatment is carried out. The effect of modification is particularly well visible when the average grain surface area and grain count are compared (Figs. 3 and 4, respectively).

Modification by the route adopted in experiment 4 (casting of 17 mm cross-section) has resulted in an almost 200 fold increase of the precipitates count and an over 40 fold decrease of their average surface area.

The morphology of these grains, and specifically the degree of their refinement, as well as the stereological parameters depend on the technique by which the inoculant is introduced to alloy (the way in which it is placed in the gating system) and on the alloy pouring temperature. Yet, the grain size alone is not enough to compare the specimens. The differences in the grain surface area described by the surface area variability coefficient (Fig. 5) are also important. The higher is the value of the variability coefficient, the larger is the grain size heterogeneity. In modified specimens, the values of this parameter are comparable, while in the non-modified specimens, they assume rather low values, which means that coarse grains of a relatively low heterogeneity are prevailing. Modification not only changes the size of the grains but also their shape (Fig. 6). The higher is the shape factor value, the more compact are the grains.

With prolonged time of contact between the liquid alloy and inoculant, this effect becomes more prominent.

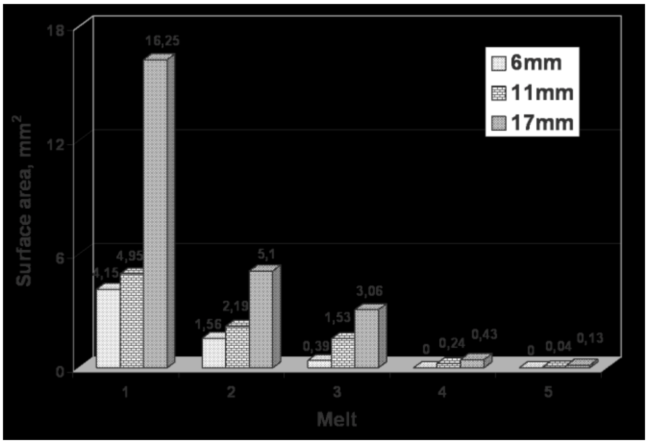

Fig. 3. A comparison of the average grain surface area

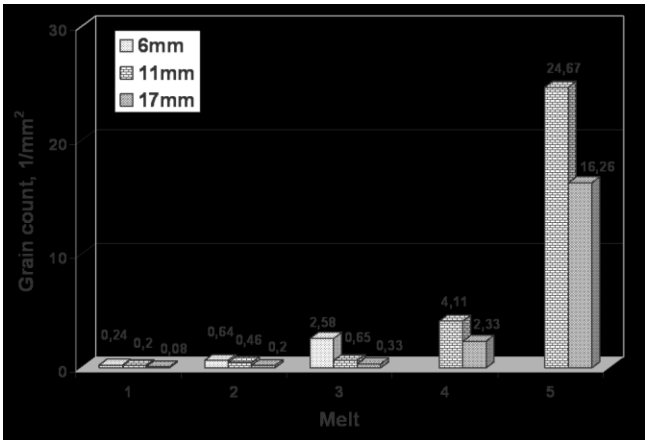

Fig. 4. A comparison of the grain count per unit surface area

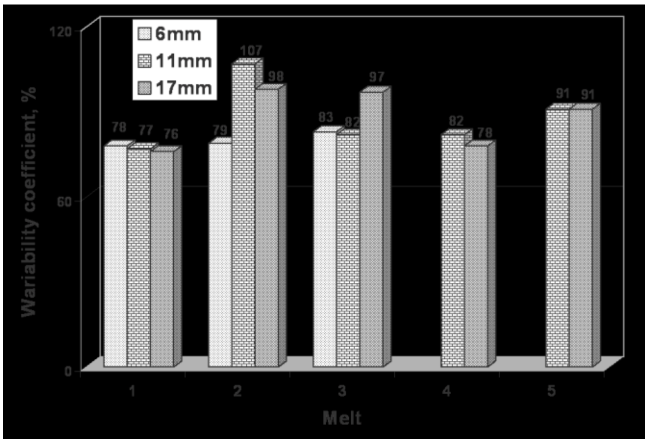

Fig. 5. A comparison of the grain surface variability coefficient

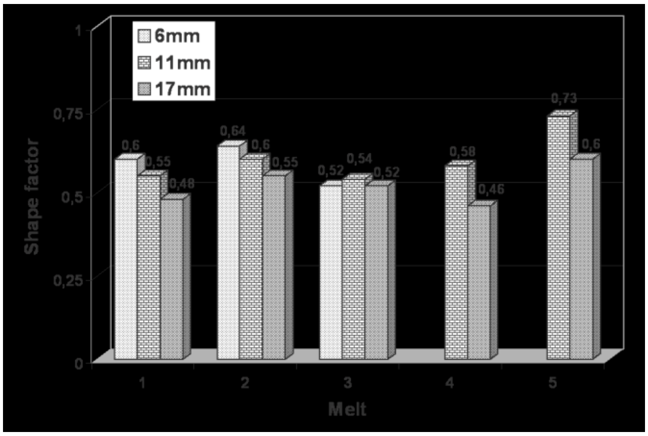

Fig. 6. A comparison of the average values of the grain shape factor

As follows from the conducted investigations, the best modification effect is obtained when alloy is poured at a temperature of up to 1440°C. The temperature of pouring (modification) raised above 1440°C definitely weakens the modification effect. An important role (maybe very important even) has the liquid alloy/inoculant contact time. The time of contact can be prolonged through proper design of a gating system. The effect of modification temperature has been proved by the results obtained in investigations of the surface modification process – the lower is the alloy temperature, the stronger is the modification effect. Yet, to preserve good alloy castability, necessary for a faithful shape reproduction (e.g. of engine blades), the pouring temperature must never go below 1440°C.

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