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# Macrostructure of IN-713C superalloy after volume modification

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#### Abstract

The study discusses the results of the preliminary investigations of the process of volume modification of nickel and cobalt superalloys. The investigations were carried out on an IN-713C alloy. As an inoculant, cobalt aluminate  $CoAl_2O_4$  in composition with aluminium powder and zircon flour was used. Experiments included the use of inoculant-coated filters and inoculant placed on and between the filters. As a criterion for evaluation of the modification effect, the macrostructure produced in a stepped test casting was used. It has been concluded that the modification effect depends, first of all, on the pouring temperature and on the time of contact between the liquid alloy and inoculant. Compared with the non-modified alloy, which has columnar grains in its structure, the refining treatment gave very fine and equiaxial grains. The higher was the cooling rate (or the smaller was the thickness of the step), the finer were the equiaxial grains.

Keywords: nickel superalloys, macrostructure, modification, columnar and equiaxial grains, pouring temperature, CoAl<sub>2</sub>O<sub>4</sub> inoculant

#### 1. Introduction

In consideration of the flight safety problems, the structural elements forming the "hot parts" of an aircraft engine are subject to quite exceptional manufacturing regime and quality control. At present, the near-net-shape castings for parts of aircraft engines are made from modern grades of nickel and cobalt alloys, to mention just the families of INCONEL 100, INCONEL 713C, RENE 77, MAR-M257 and MAR M 509 [1, 2]. These are the alloys precipitation hardened, which during solidification can form a specific macrostructure, composed of the grains equiaxial, frozen and columnar. A structure of this type is prone to crack formation and propagation, resulting in fatal failure of the aircraft engines [3, 4].

Parts cast from these alloys are, moreover, expected to offer very narrow dimensional tolerances, excellent surface quality as-cast and after heat treatment, and minimum level of gasinduced and shrinkage porosities. It is also recommended to obtain the structure of equiaxial grains within the entire casting volume.

World's technical literature provides abundant information on methods of refining the macro- and microstructure of nickel superalloys, using the technique of refining [5] and modification with nanoparticle inoculants [16-9]. In [6] the results of modification of Inconel 718 type alloy with microadditions of cobalt oxide CoO were presented. A minor degree of structure refinement and slight improvement of the mechanical properties was obtained. In [7] the authors were discussing the results of investigations on the modification of nickel superalloys with an inoculant 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 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]. It was observed that the modifying additives should have the density from 8000 to 8800 kg/m<sup>3</sup>, the melting point above 1500 °C, and the crystal lattice identical with or similar to the crystal lattice of the alloy matrix. In any case, the difference in lattice parameters should not exceed 10%. Particularly favourable results were obtained for a mixture of the Co<sub>3</sub>FeNb<sub>2</sub> and CrFeNb intermetallic phases [11].

Considering the lack of any more comprehensive information on the effect that the volume modification is expected 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.

#### 2. The research problem

The so far applied surface modification (with inoculant placed in the face layer of a ceramic mould) is not satisfactory from the designer's point of view; moreover, the mechanism by which this treatment is affecting the primary structure of castings made from the above mentioned alloys is practically unknown.

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



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

The individual elements of the stepped test piece cast in a mould without the inoculating coating reveal on the cast surface the presence of the grains much coarser than the grains observed in a casting poured into a mould coated with inoculant. Moreover, in non-modified casting, the cooling rate has no effect on the grain size. Macrostructures on cross-sections show a very superficial modification effect, in the best case penetrating inside the casting to a very small depth only. In the direction towards the casting centre, the presence of the highly undesired columnar crystals was 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 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):

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

Owing to a good agreement that exists between the crystal lattice forming the matrix of nickel and cobalt superalloys and the hightemperature Co particles obtained in the reaction of exchange (reduction), the nucleation of crystals can be initiated in the matrix.

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. Considering the fact that moulds are preheated, this time lapse can be measured quite easily. This is the reason why in thin-walled castings, cooling at a higher rate (with higher undercooling), the number of the surface nuclei that do not dissolve in liquid metal will be larger, and due to this, the effect of the surface modification will be more prominent.

Therefore it seems advisable to undertake investigations on the process of volume modification, mainly to find out a way to provoke the crystallisation of nuclei still before the liquid alloy enters mould cavity, e.g. in the gating system.

## 3. Materials and methods of investigation

Studies were carried out 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 a Balzers VSG-02 induction furnace in an  $Al_2O_3$  crucible. The charge weight was about 1,2 kg. Melting was carried out under the protective argon atmosphere. Stepped test pieces of dimensions adjusted to the size of the induction furnace chamber were cast. For investigations, the elements of the stepped test piece of 6, 11, 17 and 23mm wall thickness were used. This enabled an additional evaluation of the cooling rate effect on grain size. Before being placed in furnace chamber, the moulds were preheated to 750°C. The temperature of both the liquid metal and ceramic mould was controlled with a Pt-PtRh10 immersion thermocouple. The alloy pouring temperature was from 1400 to 1500°C.

Figure 2 shows a general view of the adapted ceramic mould, while Figure 3 shows the furnace with mould and charge inside (before closing).

The inoculants were prepared from cobalt aluminate  $CoAl_2O_4$ , aluminium powder, and zircon flour, all mixed in different ratios. As a binder, the colloidal silica was used. The product was crushed after drying. Several experiments were made, varying the temperature of pouring and the inoculant location, and changing in this way also the molten alloy–inoculant contact time. The criterion used in evaluation of the modification effect was macrostructure obtained in the stepped test pieces of 6, 11 and 17mm thickness. In the study, the results of the following experiments are presented:

1. Alloy without modification, pouring temperature of 1480°C.

2. Inoculant in the amount of 1g, placed on filter, pouring temperature of  $1500^{\circ}$ C.

3. Inoculant in the amount of 1g, placed on aluminium foil between filters, pouring temperature of 1440°C.

4. Inoculant in the amount of 1g, placed between filters, pouring temperature of  $1420^{\circ}$ C.

5. Inoculant in the amount of 0,5g, placed between filters (with side hole), pouring temperature of 1400°C.



Fig. 2. The investment ceramic moulds made from zircon flour and mullite



Fig. 3. A view of the vacuum induction furnace, model VSG-02

# 4. The results of investigations and discussion of results

The specimens for macrostructural examinations were etched with Marble's reagent. The results of the examinations obtained on specimens taken from the cast walls of 11 and 17mm thickness are shown in Figures 4 to 8. Because of difficulties in identification of the grain size after modification carried out according to variants 4 and 5, the images of macrostructure obtained in specimens taken from the 6mm cast wall were not shown.



Fig. 4. Macrostructure of specimens - experiment 1



Fig. 5. Macrostructure of specimens - experiment 2



Fig. 6. Macrostructure of specimens - experiment 3



Fig. 7. Macrostructure of specimens – experiment 4



Fig. 8. Macrostructure of specimens - experiment 5

The images of macrostructure obtained in castings made from IN-713C alloy confirm the beneficial effect of in-mould volume modification on the elimination of columnar grains and crystallisation of the equiaxial ones. The morphological features of these grains, and especially the degree of refinement as well as other stereological parameters depend on the type of the inoculant, on the way in which this inoculant is placed in mould, and on the temperature of pouring. An important technological parameter is also the liquid alloy–inoculant contact time. A prolongation of this time can be obtained through proper design of the gating system. The lower is the alloy temperature, the stronger is the modification effect, as confirmed by the conclusions drawn from surface modification. Yet, to preserve good alloy castability, required for faithful reproduction of the casting shape (e.g. in the case of engine blades), the pouring temperature should not be lower than 1400°C.

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