

Phase transformations and microstructure of IN-713C nickel superalloy

F. Binczyk*, J. Śleziona

Chair of Metal Alloys and Composites Engineering

Silesian University of Technology, Krasińskiego Str. 8, 40-019 Katowice, Poland

*Contact for correspondence: e-mail: franciszek.binczyk@polsl.pl

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Abstract

The study presents the results of investigations of phase transformations taking place during melting and solidification and of microstructural examinations carried out on the family of IN 713C nickel superalloys. Examinations were carried out by the method of thermal analysis (ATD) and differential scanning calorimetry (DSC). It has been concluded that the method of thermal analysis (ATD) enables more precise assessment of the precipitation of the primary phases of a low value of the solidification enthalpy. The advantage of DSC is the possibility of determination of the value of the heat (enthalpy) of phase transformations during alloy melting and solidification. The measured parameters of T_{ik} and T_{sol} are comparable for both methods. Microstructural examinations have confirmed the phenomena accompanying phase transformations, i.e. the precipitation of primary carbides, the solidification of γ phase matrix and carbide eutectic, and the formation of γ' phase in solid state.

Keywords: nickel superalloys, ATD analysis, DSC analysis, temperature, solidification, microstructure, carbides

1. Introduction

Creep-resistant nickel alloys are the base material used for cast parts of aircraft engines, both static parts as well as the rotating guiding elements, operating at high temperatures under the effect of mass forces [1-3]. The requirements imposed on these castings include, among others, high fatigue resistance, creep resistance at high temperatures, and resistance to corrosion in media containing products of fuel combustion. The castings are made in near-net-shape moulds by investment process. Currently, the investment castings for parts of aircraft engines are being made from the modern family of nickel and cobalt alloys, including IN-713C. Depending on the solidification conditions and modification process, the macrostructure of this alloy is composed of, present in different content ratios, equiaxial grains, frozen and columnar, with precipitates of primary carbides inside the grains and on the

grain boundaries. In structure of this type, cracks may form and result in fatal failure of the aircraft engines [4, 5].

World's technical literature provides abundant information on the methods of refining the macro- and microstructure of nickel superalloys by the technique of refining [6] and inoculation with nanoparticle inoculants [7-9].

Yet, to be able to control the microstructure formation and properties of the ready products, it is necessary to know first the phenomena that take place during solidification of these alloys. This mainly refers to the temperature of the beginning of solidification, the temperature range of the primary phases and matrix solidification, and the temperature of solid state transformations. This, in turn, enables choosing the best pouring temperature to provide sufficient alloy castability and ability to faithfully reproduce the casting configuration.

2. Materials and methods of investigation

Alloy solidification was examined by the ATD analysis; calorimetric examinations were also conducted. Studies were made 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). As expected, these constituents are responsible for the formation of alloy microstructure. The main parameters are the temperature T_{lik} and the solidification range within which the morphology of the γ matrix and carbide eutectic is developed. When in solid state, these alloys experience an important phase transformation of $\gamma \rightarrow \gamma'$.

Melts were prepared in a VSG-02 induction furnace (made by Balzers), using an Al_2O_3 crucible. The charge weight was about 1,2 kg. Melting was carried out under argon protective atmosphere. The alloy was cast into a ceramic mould at a temperature of 1540°C, producing a rod of $\phi 40 \times 60$ mm.

The solidification process was recorded by a Crystaldigraph PC-8T apparatus. Schematic representation of the test stand is shown in Figure 1.

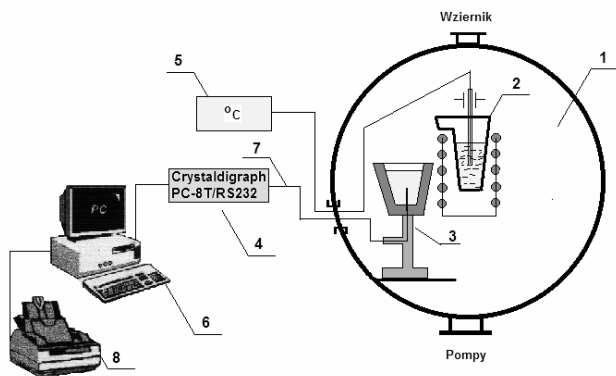


Fig. 1. Schematic representation of a test stand: 1- chamber of VSG-02 furnace; 2- crucible and induction coil; 3 – graphite foundry mould; 4 – Crystaldigraph PC-8T apparatus; 5 – digital temperature measuring unit; 6 – PC computer; 7 – compensation lead; 8 – printer

For the determination of temperature range and thermal effect of phase transformations during heating and cooling, a Multi HTC S60 differential scanning calorimeter was used. The weight of samples was similar and ranged from 310 do 340 mg. Tests were made under argon protective atmosphere at a heating and cooling rate of 10°C/min. Samples were preheated to 1450°C

3. The results of investigations

The thermal analysis curve plotted for an IN-713C alloy is shown in Figure 2. Figures 3 and 4 show plotted results of the

differential scanning calorimetry (DSC) made during heating and cooling of the specimens.

Below, typical values of the temperature taken from the plotted ATD curve are given:

- A – T_{ma} , the maximum alloy temperature, 1519°C,
- B – T_{lik} , the crystallisation temperature of γ phase, 1318°C
- D – T_E , the eutectic solidification temperature, 1284°C
- E – T_{sol} , the temperature of the end of alloy solidification, 1256°C,
- G – T_f , the temperature of solid state transformation, 1146°C

On the other hand, the characteristic temperature values taken from the DSC cooling curve are as stated below:

- T_{ma} , the maximum alloy temperature, 1425°C,
- T_{lik} , (1 top) the solidification temperature of γ phase, 1323°C
- T_E , (2 top) the eutectic solidification temperature, 1305°C
- T_{sol} , the temperature of the end of alloy solidification, 1252°C,
- T_f , the temperature of solid state transformation, 1152°C

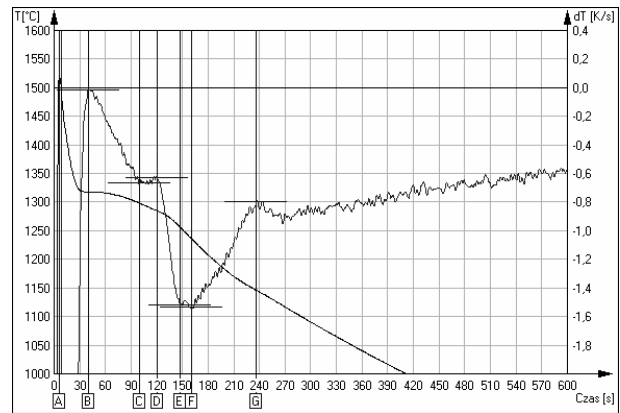


Fig.2. Plotted curve of ATD analysis and values measured at points characteristic of IN-713C alloy

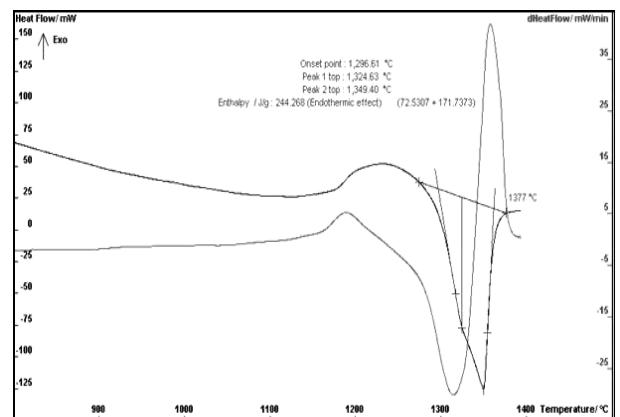


Fig. 3. The DSC diagram of IN7-13C alloy heating

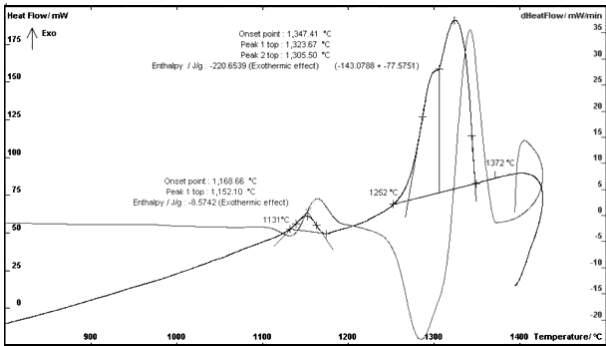


Fig. 4. The DSC diagram of IN-713C alloy cooling

An example of microstructure obtained for the examined IN 713C alloy in unetched state is shown in Figure 5. The regions of microanalysis of the matrix and carbides are shown in Figures 6 and 7, respectively. The specimen was etched with Marble's reagent.

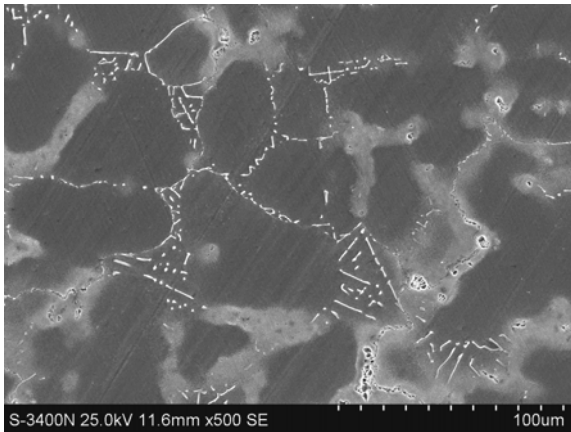


Fig. 5. Microstructure of IN-713C alloy. Note the typical "chain-like" distribution of carbides

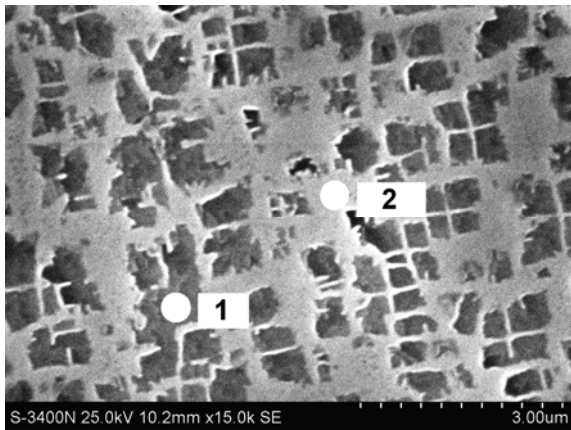


Fig. 6. Microstructure of IN-713C alloy matrix. The regions of microanalysis (dark- γ' phase, light- γ phase)

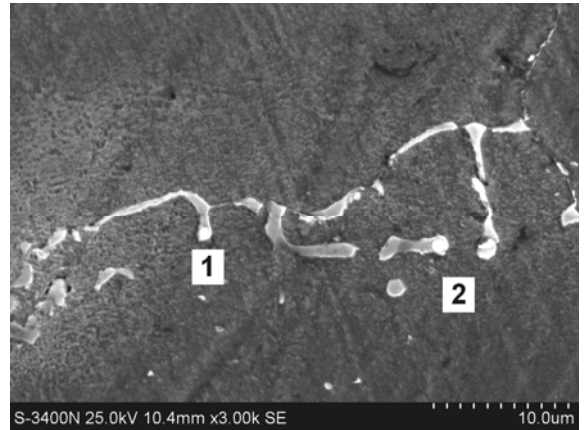


Fig. 7. The regions of microanalysis of eutectic carbides

The results of microanalysis of the main alloy constituents present in the microregions of matrix and in carbide eutectic precipitates are shown in Table 1.

Table 1. Chemical composition in microregions of the specimen

Area	% wt.						
	Al	Cr	Ti	Nb	Mo	Ni	
Matrix	1	6,11	13,94	0,93	1,74	4,73	72,49
	2	4,88	14,44	0,73	1,60	4,99	73,37
Carbides	1	1,86	6,08	7,30	51,60	11,95	21,22
	2	2,13	7,16	6,77	45,38	12,76	25,80

4. Discussion of results

Basing on the investigation results, the characteristic solidification parameters obtained by ATD and DSC were compared. The results are shown in Fig. 8.

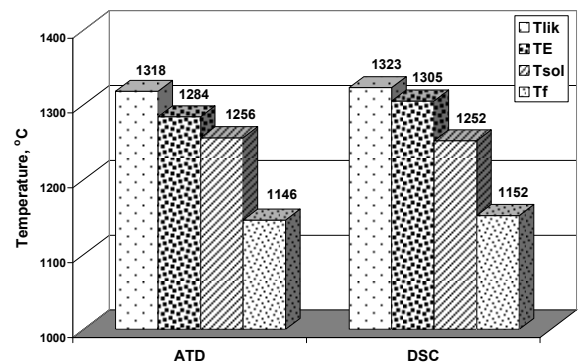


Fig. 7. A comparison of the characteristic values of the solidification temperature determined by the methods of ATD and DSC analysis

It can be concluded that the obtained values of solidification parameters are comparable, though slightly higher in the case of calorimetric method.

The microstructural examinations of IN-713C nickel alloy confirm the exothermic heat effects observed on ATD curves, due to the matrix (γ phase) and carbide eutectic crystallisation on grain boundaries, characterised by a chain-like distribution (the Chinese script). The thermal effect due to the eutectic solidification is more prominent on the ATD curve. The value of the matrix solidification enthalpy is about 143,07 J/g, and of the carbide eutectic - about 77,57 J/g.

When in solid state, the alloy undergoes a transformation of γ phase (light regions) into γ' phase (dark regions); the effect is due to structure ordering. The DSC results confirm particularly well this fact. On these curves, the exothermic effects in solid state are best visible, with the enthalpy of transformation amounting to about 8,57 J/g.

The obtained results enable drawing a conclusion that the ATD method of thermal analysis offers more possibilities for interpretation of the first stage of solidification process, when from metal in liquid state the primary phases of a low heat of solidification are precipitating. In DSC method, because of a very small weight of the samples, these effects cannot be observed.

The calorimetric method, on the other hand, seems to be more useful in investigation of the solid state phase transformations. The values of the solidification parameters (T_{lik} and T_{so}) obtained by both methods are comparable, especially during alloy solidification. The undeniable advantage of the DSC method is the possibility to measure the value of the heat (enthalpy) of phase transformations.

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