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# ATD and DSC Analysis of IN-713C and ZhS6U-VI Superalloys

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## Abstract

Paper presents the results of ATD and DSC analysis of two superalloys used in casting of aircraft engine parts. The main aim of the research was to obtain the solidification parameters, especially  $T_{sol}$  and  $T_{liq}$ , knowledge of which is important for proper selection of casting and heat treatment parameters. Assessment of the metallurgical quality (presence of impurities) of the feed ingots is also a very important step in production of castings. It was found that some of the feed ingots delivered by the superalloy producers are contaminated by oxides located in shrinkage defects. The ATD analysis allows for quite precise interpretation of first stages of solidification at which solid phases with low values of latent heat of solidification are formed from the liquid. Using DSC analysis it is possible to measure precisely the heat values accompanying the phase changes during cooling and heating which, with knowledge of phase composition, permits to calculate the enthalpy of formation of specific phases like  $\gamma$  or  $\gamma'$ .

**Keywords:** Innovative casting materials and technologies, Nickel superalloys, ATD, DSC, Solidification parameters

## 1. Introduction

Structure and phase composition of turbine blade castings have immense effect on their usability and properties [1]. Because of that good understanding of processes taking place during solidification and cooling of castings is important. In this, the various thermal analysis techniques, like thermal-derivative analysis (ATD), differential thermal analysis (DTA) or differential scanning calorimetry (DSC) are useful tools.

Phase transitions are an exothermic and endothermic processes and thus can be observed by precise temperature measurements [2-4]. The differential techniques are more complex however tend to be more precise than derivative analysis, for example the DSC allows for more precise observation of solid state phase transitions [5-7].

The most commonly used solidification parameters characteristic for any casting alloy are solidus ( $T_{sol}$ ) and liquidus

( $T_{liq}$ ) temperatures.  $T_{liq}$  is the temperature in which the very first solid crystals are formed in the molten metal and is useful in optimisation of the pouring temperature to ensure the good balance between liquid metal fluidity, casting shrinkage and solubility of impurities.  $T_{sol}$  temperature is useful in design as a limiter of operation temperature and for optimisation of heat treatment processes [8-11].

Depending of alloy composition, varied eutectics can form during solidification. The low-melting point eutectics precipitating in the last stages of solidification may have adverse influence on high-temperature properties of the alloy especially when high-temperature heat treatment is used because during such procedures these eutectics melt and their constituents are dissolved into the alloy matrix leading to dangerous increase in micro-porosity [12].

The formation of low melting point eutectics often imply impurity of the alloy. Presence of these phases can be detected using thermal analysis. In ATD analysis the presence of low-

melting point eutectics significantly lengthens the last stage of solidification and lower the value of  $dT/dt$  derivative. It also changes the inclination of the tangent to the derivative line on the ATD graph [13]. Sample situation is presented in Fig. 1.

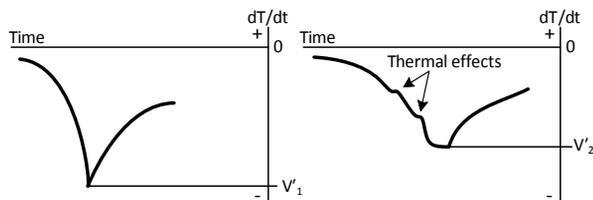


Fig. 1. ATD graph for the ending of solidification for high quality alloy (left) and low quality alloy (right)

## 2. Materials and methods of investigation

Research was carried out using ZhS6U-VI and IN-713C (from different delivery batches) that were provided by Pratt & Whitney Rzeszow foundry.

Correct interpretation of ATD data is possible only when the conditions of the test are kept the same in all cases. This was ensured by using as follows:

- ceramic test moulds of same dimensions and material,
- precise, good quality type S thermocouples,
- closely matched quantities of tested alloy ( $\approx 800$  g),
- closely matched pouring temperatures ( $\approx 1500$  °C),

The test casting for ATD analysis was designed as a  $\varnothing 30 \times 120$  mm rod with  $40 \times 45 \times 17$  mm riser. The temperature was measured by type S thermocouple shielded with quartz glass and placed at 1/3 of the casting height (from the base).

Melting was carried out using the Balzers VSG-02 VIM furnace in a  $Al_2O_3$  crucible. The charge was about 1200 g. Protective atmosphere of Ar gas at a pressure of  $9 \cdot 10^{-4}$  Pa was used during melting process. The temperature of molten metal was measured by a type S thermocouple immersed in the crucible. The ceramic mould placed in a VIM furnace chamber is shown in Fig. 2.



Fig. 2. Ceramic mould in a furnace chamber

Calorimetric analysis was carried out using Multi HTC S60 differential scanning calorimeter. The weight of samples varied from 140 to 250 mg. Tests were carried out under protective argon atmosphere with heating and cooling rate of  $10$  °C / min. Preheating temperature was set to  $1450$  °C.

Knowledge about the metallurgical quality of feed ingots before production allows to greatly reduce the share of faulty castings. Feed ingots may be contaminated by oxides present in

shrinkage cavities formed during preparation of the ingot and, because the superalloy ingots are cast in shape of long rods, the concentration of impurities is not homogeneous. Schematic representation of the phenomena and the photography of actual cross section of the ingot are shown in Fig. 3.

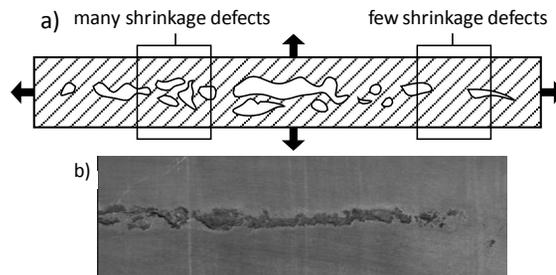


Fig. 3. Shrinkage cavities in feed ingots: a) schematic, b) actual defects on the cross section of the ingot

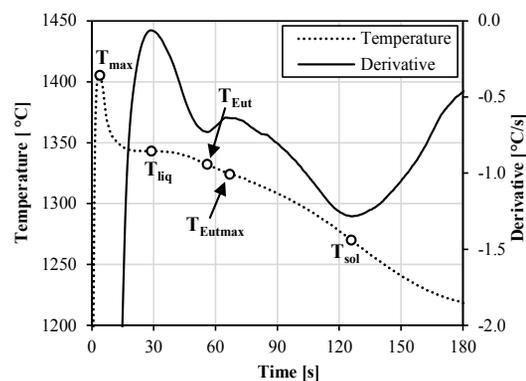
During previous ATD analysis of IN-713C [8,10,13] it was found that good metallurgical quality is indicated by:

- $T_{liq}$  higher than  $1335$  °C,
- $T_{sol}$  higher than  $1250$  °C,
- low ( $T_{Eut} - T_{sol}$ ) difference, ordinarily lower than  $60$  °C,
- low ( $t_{sol} - t_{Eut}$ ) time range, ordinarily lower than  $50$  s,
- absence of abnormal thermal effects,
- high derivative  $V'$  (°C·s<sup>-1</sup>), ordinarily above  $1.5$  °C·s<sup>-1</sup>.

Thermal properties of ZhS6U-VI alloy are similar [14,15] therefore the aforementioned indicators are valid for both alloys.

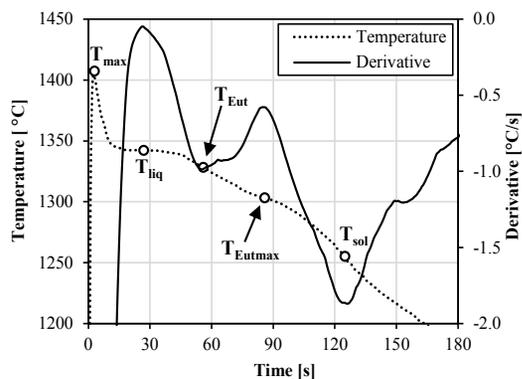
## 3. The results of investigations and discussion of results

ATD graphs and characteristic temperature values register in the course of the experiments are shown in Figs. 4, 5 and 6.



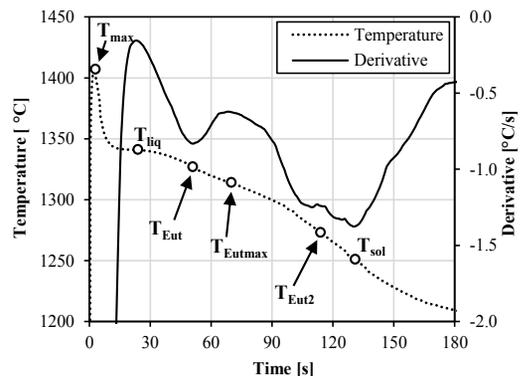
Parameter	Time [s]	Temperature [°C]
$T_{max}$	4	1405
$T_{liq}$	29	1343
$T_{Eut}$	56	1332
$T_{Eutmax}$	67	1324
$T_{sol}$	126	1270

Fig. 4. The ATD analysis of ZhS6U-VI alloy



Parameter	Time [s]	Temperature [°C]
$T_{max}$	3	1407
$T_{liq}$	27	1342
$T_{Eut}$	56	1328
$T_{Eutmax}$	86	1303
$T_{sol}$	125	1255

Fig. 5. The ATD analysis of IN-713C alloy (batch 4V5910)



Parameter	Time [s]	Temperature [°C]
$T_{max}$	3	1407
$T_{liq}$	24	1341
$T_{Eut}$	51	1327
$T_{Eutmax}$	70	1314
$T_{Eut2}$	114	1273
$T_{sol}$	131	1251

Fig. 6. The ATD analysis of IN-713C alloy (batch 6V8173)

Data obtained during ATD analysis was used to assess the metallurgical quality of selected alloys:

#### Alloy ZhS6U-VI

1. Low value of  $T_{liq} = 1343\text{ °C}$
2. High value of  $T_{sol} = 1270\text{ °C}$
3. Significant difference ( $t_{sol} - t_{Eutmax}$ ) = 57 s.
4. Significant difference ( $T_{Eut} - T_{sol}$ ) = 54 °C.
5.  $V' = 1.28\text{ °C}\cdot\text{s}^{-1}$ .

Graph is acceptable, especially in the last stage of solidification (no additionally heat effects), which signifies **good** metallurgical quality of the alloy.

#### Alloy IN-713C (batch 4V5910)

1. High value of  $T_{liq} = 1342\text{ °C}$ .
2. Lower value of  $T_{sol} = 1255\text{ °C}$  (compared to Zh6U-VI alloy).

3. Small difference ( $t_{sol} - t_{Eutmax}$ ) = 39 s.
4. Small difference ( $T_{Eut} - T_{sol}$ ) = 48 °C,
5.  $V' = 1.88\text{ °C}\cdot\text{s}^{-1}$ .

Graph is acceptable, especially in the last stage of solidification (no additionally heat effects), which signifies **very good** metallurgical quality of the alloy.

#### Alloy IN-713C (batch 6V8173)

1. High value of  $T_{liq} = 1341\text{ °C}$ ,
2. Low value of  $T_{sol} = 1255\text{ °C}$  (compared to Zh6U-VI alloy),
3. Large difference ( $t_{sol} - t_{Eutmax}$ ) = 61 s,
4. Large difference ( $T_{Eut} - T_{sol}$ ) = 63 °C,
5.  $V' = 1.36\text{ °C}\cdot\text{s}^{-1}$ .
6. Additional heat effect from oxide eutectic!

Graph, especially in the last stage of solidification (additional heat effects), signifies **low** metallurgical quality of the alloy.

Sample DSC analysis data for ZhS6U-VI alloy obtained during heating and cooling are presented in Figs. 7 and 8.

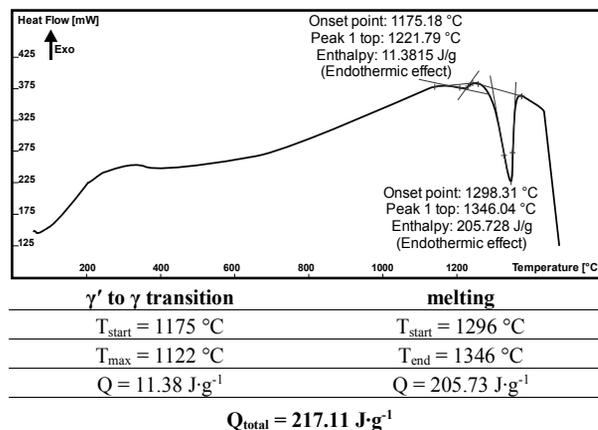


Fig. 7. The DSC diagram of ZhS6U-VI alloy heating

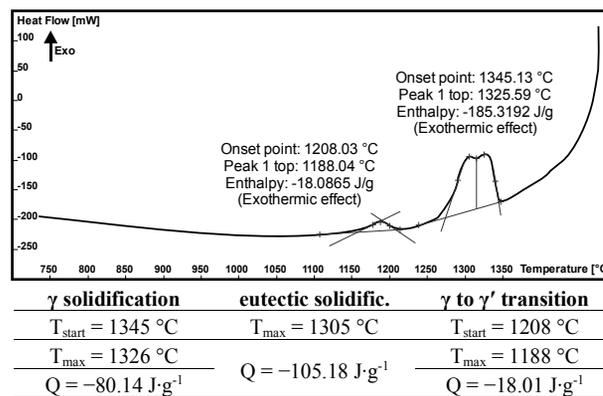


Fig. 8. The DSC diagram of ZhS6U-VI alloy cooling

The DSC data for IN-713C alloy samples were similar to presented results for ZhS6U-VI alloy. Values of characteristic temperatures are higher during melting than during solidification which is caused by overheating and overcooling.

Energy balance of  $\gamma$  to  $\gamma'$  (cooling) and  $\gamma'$  to  $\gamma$  (heating) transitions indicates smaller initial share of  $\gamma$  phase in the

samples. The total heat value change during solidification is less than this value during melting (which is normal because of the energy losses).

A more detailed analysis of the DSC results requires knowledge of the structural composition of the samples before and after the tests.

The ATD results show that this method is exceptionally useful for detecting a low-heat primary phase transitions at the beginning of solidification where, especially in comparison to DSC analysis in which small weight of the samples makes these phenomena hard to notice.

On the other hand, the precision of DSC analysis allows to better interpret the solid-state phase transitions which are common for nickel superalloys (most of all the order – disorder,  $\gamma$  to  $\gamma'$  transition)

The determined values of characteristic solidification parameters ( $T_{liq}$  and  $T_{soi}$ ) are comparable between both the ATD and DSC analysis. However during the calorimetric analysis, it is also possible to calculate the enthalpy of phase transitions.

## Acknowledgements

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