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DETERMINATION OF THERMOPHYSICAL AND STRUCTURAL PROPERTIES OF NICKEL SUPER-ALLOY

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In this work the differential thermal analysis (DTA) was selected for the study of 718Plus super-alloy. Particular attention was paid to determination of the phase transformation temperatures (liquidus, γ´ precipitation temperature, etc.). Almost at all temperatures of samples an undercooling was observed. Shifting of almost all temperatures was observed in the heating/cooling mode towards higher values with an increasing rate of heating, lower values with the increasing cooling rate. On the basis of DTA and structural analysis it may be stated that development of phase transformations will probably correspond to the following scheme: melting → γ phase; melting → γ + MC (NbC, TiC); melting + MC → γ + Laves + δ; γ → γ´ (γ´´).

Key words: Ni super-alloy, differential thermal analysis, thermophysical properties, structural properties

INTRODUCTION

Differential thermal analysis (DTA) [1] is dynamic thermal-analytical method, which monitors thermal effects of investigated sample, connected with its physical and chemical changes at its continuous, linear heating or cooling. With use of this method it is possible to obtain temperatures and latent heats of phase transformations.

Nickel super-alloys are class of alloys containing an atomic concentration greater than 50 % of nickel and having excellent physical properties, such as strength, hardness and creep resistance, at the temperatures close to their melting points. The exact physical properties are a result of different alloying elements and thermal treatments, which made them suitable for high temperature applications in turbine engines for power generation, gas turbine jet engines for aircraft, and for components in the space technology and energy production [2].

For these reasons it is necessary to pay attention to acquisition of reliable material data, which are needed for modelling of processes, for control of solidification processes, but also for improvement of process procedures and for enhancement of their efficiency. Typical necessary data are temperatures of phase transformations, latent heats of phase transformations, surface tensions and other important data [3].

Although material data (e.g. temperatures of phase transformations, latent heats) were measured also for some super-alloys, e.g. [4], so far no accessible database of thermo-physical data of these systems exists.

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MATERIAL AND EXPERIMENTAL METHODS

The nickel super-alloy 718Plus, which was chosen as experimental material, is one newly developed nickel super-alloys [2]. The alloy 718Plus was developed by the company Allvac and its working temperature exceeds that of the alloy IN 718 by more than 50 °C. The alloy 718Plus is hardened by precipitation of the phase γ´ and it achieves excellent strength and creep resistance up to the temperature of 700 °C [2].

Studied material, i.e. the alloy 718Plus (Table 1) was cast by the company První brněnská strojírna (PBS) Velká Bíteš, a.s. For the purposes of measurement of thermophysical data of the alloy 718Plus the samples were not heat treated, they were used in as-received state. A bar with diameter of approx. 3 mm was mechanically cut from the casting of the alloy 718Plus and 5 samples with the height of approx. 3 mm and mass max. 200 mg were cut from it.

Table 1 **Chemical composition of nickel super-alloy 718Plus, concentration of elements / wt. %**

Cr	Fe	Co	Nb	Mo	Al	W
19,96	9,40	8,50	5,5	2,77	1,35	1,14
Ti	C	Si		Mn		B
0,77	0,039	< 0,035		< 0,01		0,004

DTA method

Method of differential thermal analysis (DTA) [1] was used for the purposes of experimental measurements. Data were acquired with use of experimental laboratory equipment for thermal analysis Setsys 18$_{TM}$ (SETARAM) and measuring rod TG/DTA of the type „S“ (S - type rod Pt/PtRh 10 %; 20 – 1 600 °C). Samples of the alloy 718Plus were analysed in corundum (Al_2O_3)

crucibles with volume of 100 μl. During heating and cooling a permanent dynamic atmosphere was maintained, Ar (> 99,9999 %). The samples of the alloy 718Plus were during experiment control heated at the rates of 1, 5, 7, 10 and 20 °C·min^{-1} within the temperature range from 20 to 1 400 °C, the samples were after reaching the temperature of 1 400 °C (after melting) control cooled by the above mentioned rates down to 20 °C.

Phase analysis

The samples before and after DTA analysis were also subjected to the phase analysis with use of scanning electron microscopy on the microscope JEOL JSM-6490LV equipped with energy dispersive analyser EDAX (EDS INCA x-act). Documentation of the microstructure was made in the mode of secondary (SEI) and backscattered (BEI) electron imaging.

Individual minority phases were identified on the basis of comparison of results of semi-quantitative EDX analysis with the published data on composition of phases occurring in the given type of nickel based super-alloy. Semi-quantitative X-ray micro-analysis was performed only in the case of particles larger than 1μm, when results were not significantly distorted by an X-ray signal from surrounding matrix.

RESULTS AND DISCUSSION OF EXPERIMENTAL MEASUREMENTS

It is possible to obtain the investigated temperatures of phase transformations from the so called DTA curves, which demonstrate heat phenomena during linear heating and cooling of the samples.

All temperatures of phase transformations are for transparency plotted next to the DTA–curve of the alloy 718Plus obtained at the heating rate of 10 °C·min^{-1} (Figure 1), and they are given in Tables 2 and 3. All temperatures of phase transformations (heating and cooling mode) were adjusted to the temperature of melting of pure Au (5N) and Ni (5N); they are given in Tables 2, 3, 4 and 5.

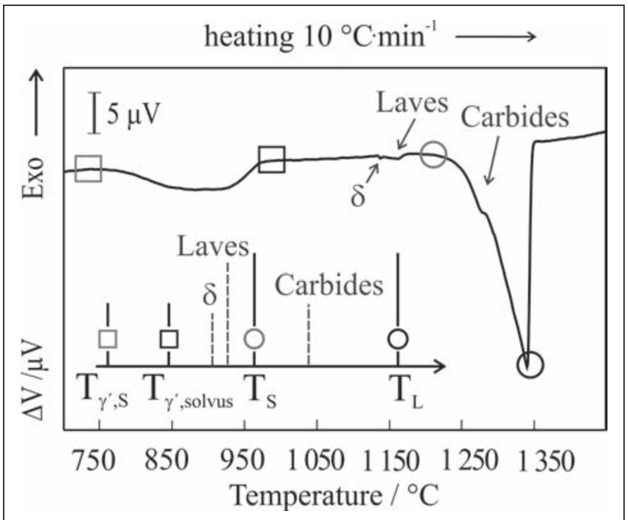

Figure 1 DTA – curve of the alloy 718Plus (heating rate 10 °C·min^{-1})

It must be noted that for the heating mode it is very difficult to determine unequivocally the temperature of start of some peaks (e.g. temperature of dissolution/precipitation of the phase γ´), for this reason temperatures of phase transformations are generally given in temperature interval. Due to the fact that final temperatures of phase transformations are also initial temperatures of the following phase of transformation, these temperatures are in the Table 2 given in one column, for example $T_{\delta,E}$ ($T_{Laves,S}$).

During the controlled heating the following temperatures of phase transformations were determined (Figure 1, Tables 2 and 3): $T_{\gamma',S}$ – initial temperature of dissolution of the phase γ´; $T_{\gamma',E}$ – final temperature of dissolution of the phase γ´; $T_{\delta,S}$ – initial temperature of dissolution of the phase δ; $T_{\delta,E}$ ($T_{Laves,S}$) – final temperature of dissolution of the phase δ (initial temperature of dissolution of the Laves phase); $T_{Laves,E}$ – final temperature of dissolution of the Laves phase; T_S – solidus temperature; T_{MC} – temperature of dissolution of MC type carbides; T_L – liquidus temperature.

Table 2 **Transformation temperatures of 718Plus, at heating**

heating rate	$T_{\gamma',S}$	$T_{\gamma',E}$	$T_{\delta,S}$	$T_{\delta,E}$ ($T_{Laves,S}$)	$T_{Laves,E}$
/ °C·min^{-1}	heating / °C				
20	830	984	1 136	1 148	1 181
10	743	990	1 136	1 145	1 179
7	772	987	1 137	1 144	1 179
5	758	987	1 137	1 143	1 179
1	733	974	1 127	1 140	1 175
0 (calc.)	727	982	1 132	1 141	1 176
10 [5]	~	936	~	~	1 167

Table 3 **Transformation temperatures of 718Plus, at heating (continuation)**

heating rate	T_S	T_{MC}	T_L
/ °C·min^{-1}	heating / °C		
20	1 198	1 286	1 346
10	1 216	1 283	1 343
7	1 224	1 282	1 341
5	1 217	1 282	1 341
1	1 220	1 274	1 340
0 (calc.)	1 226	1 277	1 339
10 [5]	1 235	1 267	1 345

During the controlled cooling the following temperatures of phase transformations were determined (Tables 4 and 5): T_L; T_{MC} – temperature of formation of MC type carbides; T_S ($T_{Laves,S}$) – solidus (initial temperature of formation of the Laves phase); $T_{Laves,S}$ – final temperature of formation of the Laves phase; $T_{\delta,S}$ – initial temperature of formation of the phase δ; $T_{\delta,E}$ – final temperature of formation of the phase δ; $T_{\gamma',S}$ – initial temperature of precipitation of the phase γ´, $T_{\gamma',E}$ – final temperature of precipitation of the phase γ´.

Almost at all temperatures of phase transformations of samples of the alloy 718Plus an undercooling was observed. Difference between the temperatures obtained at heating and cooling modes at liquidus temperatures is not so distinct (max. 46 °C) as at solidus temperatures, where undercooling may achieve even 90 °C.

From DTA curves obtained during controlled cooling by the above mentioned rates an influence of undercooling on the samples of the alloy 718Plus was observed.

Table 4 **Transformation temperatures of 718Plus, at cooling**

cooling rate	$T_{\gamma',E}$	$T_{\gamma',S}$	$T_{\delta,E}$	$T_{\delta,S}$	$T_{Laves,E}$
/ °C·min^{-1}	cooling / °C				
20	744	902	1 060	1 108	1 135
10	769	876	1 060	1 100	1 143
7	760	897	1 065	1 103	1 143
5	744	872	1 086	1 124	1 148
1	707	866	1 076	1 126	1 126
0 (calc.)	733	868	1 079	1 121	1 138
15 [6]	~	~	1 075	~	1 160

Table 5 **Transformation temperatures of 718Plus, at cooling (continuation)**

cooling rate	T_S ($T_{Laves,S}$)	T_{MC}	T_L
/ °C·min^{-1}	cooling / °C		
20	1 161	1 241	1 308
10	1 156	1 241	1 297
7	1 134	1 241	1 307
5	1 135	1 232	1 320
1	1 180	1 234	1 314
0 (calc.)	1 154	1 234	1 314
15 [6]	1 230	1 265	1 340

Apart from influence of the heating/cooling mode on shifting of temperatures of phase transformations, an influence of rate of heating and cooling on shifting of these temperatures was observed; see Tables 2, 3, 4 and 5. Shifting of almost all temperatures of phase transformations was observed in the heating mode towards higher values with an increasing rate of heating. It is evident, that rate of cooling has also influence on shifting of some transformation temperatures, and namely that temperature of the given transformation is lower with the increasing cooling rate.

On the basis of experimentally obtained temperature, the values of temperatures at zero heating (cooling) rate, '0 (calc.)', were obtained by extrapolation to the zero heating (cooling) rate (Tables 2, 3, 4 and 5).

Apart from the temperatures of phase transformations latent heat of melting 165 J·g^{-1} and latent heat of solidification (185 J·g^{-1}) was calculated on the basis of enthalpy calibration (measuring speed of 7 °C·min^{-1}). The authors [6] report for heat of solidification of the alloy IN 718 the value of 241 ± 48 J·g^{-1}.

Experimentally obtained latent heat of melting had lower value than the latent heat of solidification. The increased value of heat at cooling is probably due to the accumulation of heat in the sample during cooling. The accumulated heat is released from the sample when only after a certain energy boundary (temperature) is exceeded, when the system is already no longer able to exist in its current form, and at this moment almost immediate solidification of the sample takes place, but at lower temperature.

Analysis of the alloy 718Plus by electron microscopy

Micro-structure of the sample in as-received state and of all the samples after controlled crystallisation was qualitatively identical, Figure 2.

Four principal intermetallic phases exist [7]: Laves, γ', γ'' a δ, in γ matrix of the 718Plus alloy. Particles of γ'' phase are too small to be viewed by light of scanning electron microscopy. Small amount of elongated δ phase particles is useful on grain boundaries. In interdendritic spaces and similarly as along the grain boundaries, the carbide particles of the MC type segregated (NbC, TiC). Occurrence of sulphides, or other minority phases was not found in the samples after DTA analysis.

CONCLUSIONS

The paper investigates the temperatures of phase transformations at the precisely defined heating and cooling rates that were experimentally investigated in real samples of the nickel super-alloy 718Plus.

On the basis of the obtained values the effect of the heating/cooling rate on these temperatures was assessed. It was determined that influence of the heating and cooling rate is considerable, namely at cooling. Data obtained in the prepared database may be further used as critical input parameters for mathematical models in the area of solidification and casting of these materials.

On the basis of DTA analysis and phase analysis it may be stated that development of phase transforma-

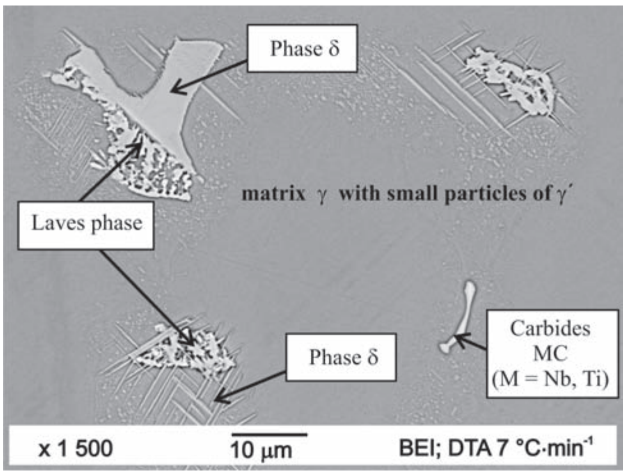

Figure 2 BEI micrograph of 718Plus, cooling rate 7 °C·min^{-1}

tions of the alloy 718Plus will probably correspond to the following scheme:

melting → γ phase; melting → γ + MC (NbC, TiC); melting + MC → γ + Laves + δ; γ → γ´ (γ´´).

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