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ISSN 0543-5846
METABK 51(1) 83-86 (2012)
UDC – UDK 669.18:621.746.32:542.63:621.22.015.5:519.651=111

THERMOPHYSICAL AND STRUCTURAL STUDY OF IN 792-5A NICKEL BASED SUPERALLOY

Received – Prispjelo: 2010-12-27
Accepted – Prihvaćeno: 2011-02-03
Preliminary Note – Prethodno priopćenje

The presented paper deals with study of phase transformations temperatures of nickel based superalloy IN 792-5A with application of DTA – method and use of experimental laboratory system for simultaneous thermal analysis SETARAM Setsys 18_{TM}. Samples taken from as-received state of superalloy were heated with controlled ramp rates (1, 5, 10 and 20 °C·min^{-1}) and immediately after melting they were cooled with the same controlled ramp rate. The samples before and after DTA-analysis were also subjected to the phase analysis with use of scanning electron microscopy on the microprobe (JCXA 733) equipped with energy dispersive analyser EDAX (EDAM 3).

Key words: DTA – method, IN792-5A, temperatures of phase transformations, scanning electron microscopy

Termofizikalna i strukturalna studija IN 792-5A niklove superlegure. Rad daje studiju temperature faznih transformacija niklove superslitine IN792-5A primjenom DTA metode te eksperimentalnog laboratorijskog sustava za simulaciju termalne analize SETARAM Setsys 18_{TM}. Uzorci su uzeti iz početnog stanja legure i održavani definiranim brzinama (1, 5, 10 i 20 °C·min^{-1}) i poslije ohlađeni kontroliranim brzinama. Uzorci su prije i poslije DTA analiza bili predmet fazne analize s primjenom skeninga elektronske mikroskopije (JCXA 733) dopunjeno s energi disperznom analizom EDAX (EDAM 3).

Ključne riječi: DTA-metoda, IN792-5A, temperature fazne transformacije, skening elektronske mikroskopije

INTRODUCTION

The aircraft industry often uses nickel super-alloys for blades of the jet-turbine engines. It is so because this material must satisfy numerous extreme requirements, such as e.g. heat-resistance at high temperatures, resistance to fatigue damage, resistance to aggressive effect of flue gases, etc. For these reasons it is necessary to pay attention to acquisition of reliable 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. Although material data (e.g. temperatures of phase transformations, latent heats) were measured also for some super-alloys, so far no accessible database of thermophysical data of these systems exists [1, 2].

Typical necessary data are temperatures of phase transformations [3-6], latent heats of phase transformations [4], specific heats [2], surface tensions [7, 8] and other important data (thermal conductance, etc.). The obtained data should be used like input data for many simulation programs (simulation of the temperature and concentration fields in castings), numerical [9, 10], physical [11, 12] models and requirements of practice (casting conditions) and they should contribute to explanation of mechanism of phase transformations of nickel super-alloys, which appear to be much more complex that has been referred so far.

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DIFFERENTIAL THERMAL ANALYSIS (DTA)

Experiment

Cast poly-crystalline nickel alloy IN 792-5A strengthened by precipitation was chosen as experimental material. Material IN 792-5A was re-melted in vacuum and cast by the company PBS Velká Bíteš, a.s. by investment casting into the mould. For the purposes of measurement of thermo-physical data of the alloy IN 792-5A the samples were not heat treated, they were used in as-received state. Chemical composition (wt. %) of the cast nickel super-alloy IN 792-5A is given in Table 1.

Method of differential thermal analysis (DTA) [13] was used for the purposes of measurement of temperatures of phase transformations of the nickel super-alloy IN 792-5A. A rod with diameter of approx. 3 mm was mechanically cut from the casting of the alloy IN 792-5A and 4 samples with the height of approx. 3 mm and mass from 160 to 200 mg were cut from it.

Data were acquired with use of experimental laboratory equipment for thermal analysis Setsys 18_{TM} made by SETARAM. Samples of the alloy IN 792-5A were analysed in corundum (Al_2O_3) crucibles with volume of 100 µl. During heating/cooling a permanent dynamic atmosphere was maintained – flow rate of Ar (> 99,9999 %) was 2 l·h^{-1}. The samples of the alloy IN 792-5A were during experiment control heated at the rates of 1, 5, 10 and 20 °C·min^{-1} within the temperature range from 20 – 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.

Table 1 **Chemical composition of nickel-base super-alloy IN 792-5A / wt. %**

	Concentration of elements / wt. %								
IN 792-5A	Ni	Cr	Mo	Co	W	Al	Ti	Nb	Ta
	base	12,50	2,15	8,71	4,50	3,60	3,75	0,08	4,15
	Fe	C	Mn	Si	Cu	Zr	B	P	S
	< 0,01	0,081	0,03	0,06	–	0,02	0,014	0,003	0,003

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 samples. These curves are registered and evaluated on computer by the program SETSOFT. Figures 1 and 2 show for illustration DTA – curves obtained at heating and cooling of the samples at the rate of 10 °C·min^{-1} with marking of characteristic temperatures of phase transformations.

The same method was used for obtaining the temperatures of phase transformations at all heating/cooling rates: 1, 5, 10 and 20 °C·min^{-1}. For clearness and for better assessment of the effect of varying heating/cooling rate on shifting of transformation temperatures all the obtained DTA – curves of the alloy IN 792-5A were brought for each thermal mode into one common image (Figure 3 – heating; Figure 4 – cooling). The obtained values of temperatures were adjusted to the temperature of melting of pure Au (5N) and Ni (5N) and they are presented in Tables 2 and 3.

The temperatures for the mode of heating were read from the left to the right and for cooling from the right to the left. It must be noted that in the modes of heating and cooling it is very difficult to read unequivocally the temperature of start of some peaks (temperatures of dissolution/precipitation of the phase γ´ – $T_{\gamma'}$, temperatures of dissolution and formation of eutectics γ/γ´, temperatures of dissolution/formation of carbides MC) – for this reason these temperatures are generally given in a temperature interval [2].

Due to the need of assessment of the effect of varying heating/cooling rate on shifting of temperatures (extrapolation of temperatures to the zero heating/cooling rate) the temperatures were not summarised in the Tables as temperature intervals, but as temperatures of starts (S = Start, e.g. $T_{\gamma/\gamma',S}$), ends (E = End, e.g. $T_{\gamma/\gamma',E}$), or they were marked with subscripts 1 - 4 (e.g. $T_{MC,1}$). Due to the fact that interval of temperature of solidus and dissolution (formation) of eutectics γ/γ´are identical, they are given in Tables 2 and 3 in one column, e.g. T_S ($T_{\gamma/\gamma',S}$) or T_S ($T_{\gamma/\gamma',E}$). Due to the fact that structure of the alloy IN 792-5A may contain carbides of the type

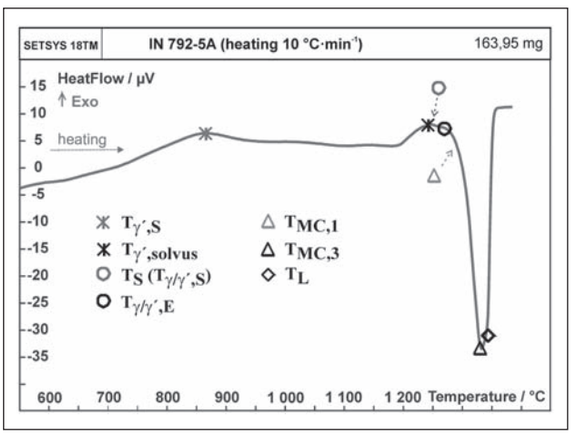

Figure 1 DTA – curve of the alloy IN 792-5A (heating rate 10 °C·min^{-1}) with marking of characteristic temperatures

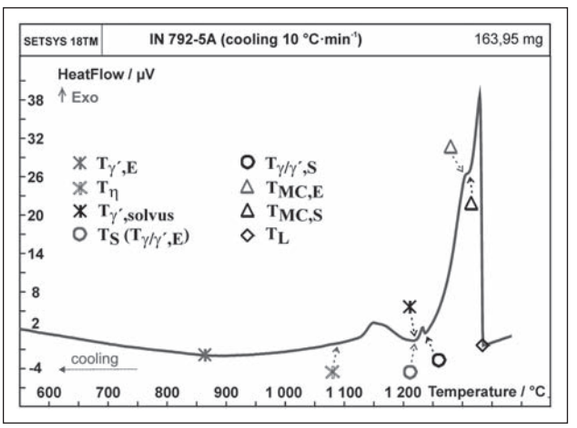

Figure 2 DTA – curve of the alloy IN 792-5A (cooling rate 10 °C·min^{-1}) with marking of characteristic temperatures

MC of various chemical composition, e.g. TaC, TiC, they are probably being dissolved progressively. Carbides will probably dissolve at various temperatures within the range of temperatures of solidus and liquidus. For this reason the temperatures are marked (Table 2): $T_{MC,1}$, $T_{MC,2}$, $T_{MC,3}$ and $T_{MC,4}$.

During the controlled heating the following temperatures of phase transformations were determined (Fig-

Table 2 **Transformation temperatures of the alloy IN 792-5A for various heating rates**

Heating rate	Temperature of phase transformation								
	$T_{\gamma',S}$	$T_{\gamma',solvus}$	T_S ($T_{\gamma/\gamma',S}$)	$T_{\gamma/\gamma',E}$	$T_{MC,1}$	$T_{MC,2}$	$T_{MC,3}$	$T_{MC,4}$	T_L
/ °C·min^{-1}	/ °C								
20	897	1 240	1 240	1 268	1 284	–	–	1 338	1 344
10	863	1 241	1 242	1 263	1 286	–	1 325	–	1 337
5	844	1 236	1 245	1 263	1 288	–	1 323	–	1 336
1	857	1 234	1 258	1 273	1 284	1 309	1 322	1 333	1 338
0 (calc.)	*843*	*1 235*	*1 254*	*1 268*	*1 286*	–	–	–	*1 336*

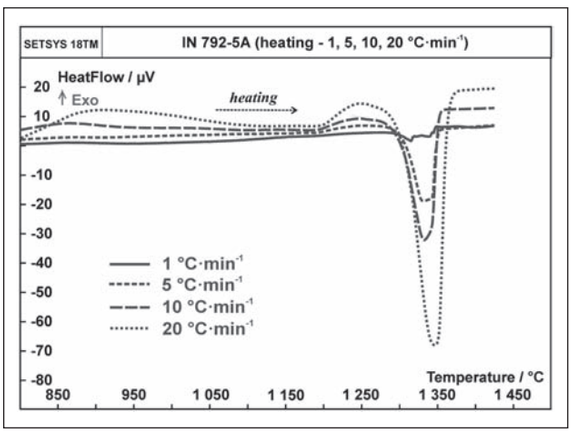

Figure 3 Evaluated DTA – curves of the alloy IN 792-5A (heating rates 1, 5, 10 and 20 °C·min^{-1})

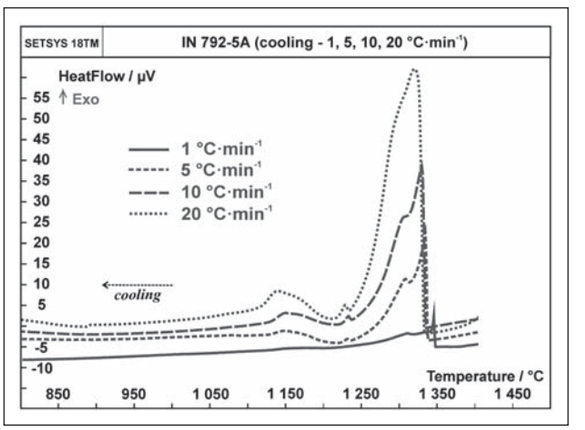

Figure 4 Evaluated DTA – curves of the alloy IN 792-5A (cooling rates 1, 5, 10 and 20 °C·min^{-1})

ure 1, Table 2): $T_{\gamma',S}$ – initial temperature of dissolution of the phase γ′; $T_{\gamma',solvus}$ – final temperature of dissolution of the phase γ′ (temperature of solubility = „solvus"); T_S ($T_{\gamma/\gamma',S}$) –solidus temperature (initial temperature of dissolution of eutectics γ/γ′); $T_{\gamma/\gamma',E}$ – final temperature of dissolution of eutectics γ/γ′; $T_{MC,1}$, $T_{MC,2}$, $T_{MC,3}$ and $T_{MC,4}$ – temperatures of dissolution of MC type carbides; T_L – liquidus temperature.

During the controlled cooling the following temperatures of phase transformations were determined (Figure 2, Table 3): T_L – liquidus temperature; $T_{MC,S}$ – initial temperature of formation of MC type carbides; $T_{MC,E}$ – final temperature of formation of MC type carbides; $T_{\gamma/\gamma',S}$ – initial temperature of formation of eutectics γ/γ′, T_S ($T_{\gamma/\gamma',E}$) – solidus temperature (final temperature of formation of eutectics γ/γ′); $T_{\gamma',solvus}$ – initial temperature of precipitation of the phase γ′ (temperature of solubility = „solvus"); T_η – temperature of formation of the phase η; $T_{\gamma',E}$ – final temperature of precipitation of the phase γ′.

Interval of temperatures corresponding to precipitation of the phase γ′ probably comprises also the interval of temperatures corresponding to precipitation of the phase η. Since this is the phase in minority quantity, it is probable that the smaller thermal effect is concealed by greater phenomenon in this temperature range. It was impossible to separate these two thermal effects from each other on the basis of DTA – curves and to determine thus unequivocally the temperature of start and end of dissolution and precipitation of the phases γ′ and η. Separation (deconvolution) of peaks by suitable software would be an appropriate solution.

The obtained values of temperatures of phase transformations were extrapolated to the zero heating and cooling rate, „0 (calc.)", and they are given also in Tables 2 and 3. We can see from the DTA curves obtained during controlled cooling the influence of under-cooling on the samples of the alloy IN 792-5A, which apparently causes the shifting of some temperatures obtained at the mode of cooling to the lower values than are the values obtained at heating (Tables 2 and 3).

Under-cooling was observed particularly in the area of temperatures of dissolution and precipitation of the phase γ′, solidus and temperatures of dissolution and formation of carbides of the type MC. The phases may be formed during heating slowly, but during cooling of the sample the development of some transformations may be distorted and this may result in shifting or even merging of the peaks on the DTA – curves obtained at cooling. For this reason it would be appropriate to use the temperatures of phase transformations obtained at heating. In the case of liquidus temperature the difference between temperatures obtained at the modes of heating or cooling is not so significant as in the case of solidus temperature.

Apart from the effect of varying heating/cooling rate on shifting of transformations temperatures, the influence of the rate on shifting of these temperatures was also observed. Shift of almost all temperatures of phase transformations was observed in the mode of heating in direction towards higher values with the increasing rate of heating. The biggest shift was observed in the temperature $T_{\gamma',S}$. It is evident from Table 3 that the cooling rate influences shifting of almost all measured transformation temperatures in such a manner, that temperature decreases with the increasing cooling rate. In the work of authors [14] temperatures of phase transformations of alloy IN 792-5A were found, which were acquired during cooling

Table 3 **Transformation temperatures of the alloy IN 792-5A for various cooling rates**

Cooling rate	Temperature of phase transformation							
	T_L	$T_{MC,S}$	$T_{MC,E}$	$T_{\gamma/\gamma',S}$	T_S ($T_{\gamma/\gamma',E}$)	$T_{\gamma',solvus}$	T_η	$T_{\gamma',E}$
/ °C·min^{-1}	/ °C							
20	1 329	1 302	1 288	1 230	1 206	1 203	1 076	871
10	1 328	1 305	1 299	1 231	1 210	1 210	1 079	859
5	1 333	1 307	1 302	1 231	1 215	1 210	1 090	861
1	1 338	1 327	1 309	1 229	1 200	1 190	1 122	890
0 (calc.)	*1 336*	*1 320*	*1 309*	*1 230*	*1 207*	*1 200*	*1 111*	*876*
[14] 5 °C·min^{-1}	1 339	1 315	–	1 245	1 215	1 175	–	–

(5 °C·min^{-1}) with use of DSC (Netzsch STA409C), which are for better lucidity presented also in Table 3.

PHASE ANALYSIS OF NICKEL BASED SUPER-ALLOY IN 792-5A

Methodology of evaluation of structure of the alloy IN 792-5A

The samples before (as-received state) and after DTA-analysis were also subjected to the phase analysis with use of scanning electron microprobe JCXA 733 equipped with energy dispersive analyser EDAX (EDAM 3).

Analysis of the alloy IN 792-5A by electron microscopy and micro-analysis

Micro-structure of the sample in as-received state and of all the samples after controlled crystallisation was qualitatively identical (Figure 5): in metallic matrix γ particles of γ′ phase were segregated, in inter-dendritic spaces the formations of eutectics γ/γ′ were segregated. With the increasing cooling rate an increased occurrence of eutectic formations was observed. Share of these formations in molten and control cooled samples decreased with the decreasing cooling rate. Coarse particles of the phase γ′ segregated along the part of perimeter of eutectic formations.

In inter-dendritic spaces and similarly as along the grain boundaries, carbidic particles MC segregated, where M represents namely Ta and Ti. Share of W, Mo and Zr in these particles was small. The largest MC particles and at the same time their lowest frequency of occurrence were found in the sample, which was cooled the most slowly (1 °C·min^{-1}).

Particles of the following minority phases occurred in small quantities before the formations of eutectics γ/γ′ [15, 16]: borides of the type M_3B_2; phase η – hexagonal phase Ni_3Ti. Occurrence of sulphides, nitrides, particles $M_{23}C_6$, or other minority phases was not detected in the samples of the alloy IN 792-5A.

CONCLUSIONS

In the presented work the temperatures of phase transformations at the precisely defined heating and cooling rates were experimentally investigated in real samples of nickel super-alloy IN 792-5A. 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/cooling rate is considerable, namely at cooling.

It is evident from the obtained results that temperatures obtained experimentally in this work and temperatures published by the authors [14] (Table 3) in some cases differ substantially. In spite of the fact that some authors investigate similar issues using similar experimental equipment, it is necessary to continue further investigation and research of the area of phase transformations of nickel super-alloys. On the basis of phase/structural analysis it may be stated that development of crystallisation of the alloy IN 792-5A will probably correspond to the published data given for the nickel super-alloys of the types IN 792 [16]: melting → γ phase; melting → MC; melting → eutectics γ/γ′; melting → η + borides.

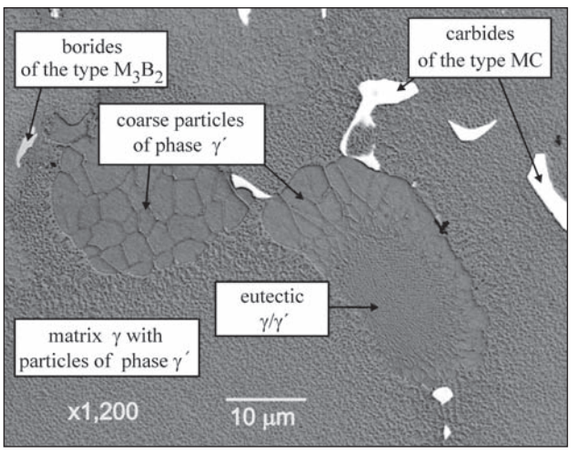

Figure 5 BEI micrograph of IN 792-5A, cooling rate 10 °C·min^{-1}

This work was carried out within the scope of the project of the Ministry of Education, Youth and Sports of the Czech Republic No. MSM6198910013.

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Note: The responsible translator for English language is B. Škandera, Dobrá, Czech Republic