

## THERMAL CONDUCTIVITY OF NICKEL SUPERALLOY MAR-M247

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Preliminary Note – Prethodno priopćenje

The paper presents the narrow connection between  $\gamma'$  phase dissolving and values of thermal conductivity. In annealing process the free space among  $\gamma'$  particles (blocks) changes in certain cycle from fine to rough and back to fine. This is accompanied by decrease and subsequent increase of thermal conductivity as well as the sample density. The results of thermal conductivity coarse are supported by image analysis.

*Key words:* nickel, superalloy, structure-phase, thermal conductivity, heat transport

### INTRODUCTION

Nickel-based superalloys are used in highly demanding applications such as energy and aerospace industries. These alloys present good corrosion resistance, weldability and mechanical stability at high temperatures. In the work [1] the differential thermal analysis was selected for the study of 718Plus superalloy.

The aim of the work [2] was to model recrystallized grain size evolution under isothermal conditions using the cellular automata technique.

Hot deformation behaviours of a typical nickel-based superalloy are investigated in [3] by isothermal compression tests under the deformation temperature range of 920 - 1 040 °C and strain rate range of 0,001 – 1 s<sup>-1</sup>. It is found that the fraction of low angle grain boundaries decreases with the increase of deformation temperature or the decrease of strain rate.

Low cycle fatigue behaviours of two cast nickel-based superalloys have been investigated in [4]. Effects of microstructure of a solution hardened alloy and a precipitation strengthened alloy on cyclic stress responses were studied at various temperatures and strain ranges than those at higher temperatures regardless of total strain range. Therefore, fatigue life of the alloy at high temperature is longer than that of low temperature under high strain range condition.

One of the most demanding applications is use of these materials for hot parts of turbines. Important position of superalloys in this area is reflected by the fact that they currently present more than 50 % of mass of modern aircraft engines [5,6]. This increase was enabled particularly by advanced processing techniques, which led to an increase of purity of alloys and thus to increase of their reliability, together with mastering of technique of directional crystallisation and subsequent technology of production of products based on single crystals.

No less important factor consisted in development of alloys with higher usable temperatures, achieved mainly by alloying, especially by Re, W, Ta and Mo [7].

In this paper we present structural influence of  $\gamma'$  phase changes on a heat transport in samples under investigation.

### THEORETICAL ASSUMPTION

The basic metal matrix of nickel superalloys is formed by the solid solution  $\gamma$  with face centred cubic lattice – FCC (face centred cubic), hardened with additive elements (cobalt, chromium, molybdenum, iron, tungsten, vanadium, titanium, niobium, aluminium, etc.) as a substitution. These elements differ in their atomic radii from the atomic radius of Ni of 1 – 13 % (13 % for W, slightly less for Mo and Cr), therefore, they can significantly harden the matrix; they also vary in the number of vacancies of electrons ( $N_v$ ), ranging from 1,0 to 7,6 (e.g. for Al,  $N_v$  is 7,6, for Ti 6,6). The  $N_v$  value is, as will be discussed below, an important parameter for determining the instability of the matrix. Alloying elements also greatly affect the mechanical and metallurgical characteristics of the basic matrix by reducing stacking fault energy (SFE). This effect increases from Ti, V, (Cr, Mo, W), Mn, Fe to Co. Low SFE leads to a reduction of the cross slip in the austenite basic matrix, and thus to an increase in its mechanical characteristics even at high temperatures.

Besides the solid solution  $\gamma$ , an essential phase in the superalloys in an amount exceeding 50 % is the  $\gamma'$  phase (Figure 1). In the first generation Ni-based superalloys, the phase occurred as globules, whereas nowadays, the  $\gamma'$  phase has a cubic shape. Chemical composition, morphology, and the distribution of the precipitate of this phase in the structure have a decisive influence on high-temperature resistance.

The  $\gamma'$  phase is a superstructure of the  $A_3B$  intermetallic compound type with  $L1_2$  lattice type – face centred cubic lattice, geometrically fully completed, where Al and Ti atoms occupy the corners of the base cell and

P. Jonšta, Vítkovice Heavy Machinery a.s., Ostrava, Czech Republic.  
P. Košťál, Z. Jonšta, T. Kulová, VŠB-TU Ostrava, FMME, Ostrava, Czech Republic. I. Vlčková, RMTSC, Material & Metallurgical Research Ltd., Ostrava, remote site VÚHŽ a.s. Czech Republic.

Ni atoms occupy space in the middle of the walls. Maintaining the same crystal lattice as with the matrix  $\gamma$  ensures the coherence of both phases. The amount of eliminated phase  $\gamma'$  and its morphology are dependent on the chemical composition of the alloy, especially on the content of Al, Ti, and also on the temperature. Generally, the higher the content of titanium and aluminium, the higher the strength of the alloys.

A measure of stability of  $\gamma'$  is most often the value of the Ti/Al ratio, considering also Nb, Ta, and Hf in addition to Ti. In many nickel alloys, the value of Ti/Al ratio is 1/1.

The heat-resistant alloys based on nickel containing carbon are always present in the structure of carbide phase. The influence of these phases on the modification of mechanical and metallurgical characteristics of superalloys is very complex. Optimal effect depends on the structure, morphology, and the distribution of the carbides, which is related to the chemical constitution of the alloy and its heat treatment. In the structure, there are four basic types of carbides, namely MC,  $M_{23}C_6$ ,  $M_7C_3$ ,  $M_6C$  [7].

Primary carbides of the MC type are usually relatively coarse, of irregular cubic morphology, and they are randomly distributed in the volume of the matrix. These carbides generally contribute very little to hardening nickel alloys.

MC carbide is formed mainly by metals, namely Ti, Ta, Nb, and V, which show a wide range of interchangeability at this stage to form combined carbides. The substitution transition of MC carbides can be prevented by increasing the content of Nb in the alloy, thereby achieving their stabilization up to temperatures ranging between 1 200 and 1 260 °C [5, 6].

## EXPERIMENTAL TECHNIQUE

Microstructural phase analysis was performed on the MAR-M247 nickel superalloy in as-cast condition and then after dissolving annealing at the temperatures of 900 °C, 1 040 °C, 1 200 °C, 1 240 °C. Table 1 gives chemical composition of the alloy.

Nickel alloys are heat treated by homogenization annealing, recrystallization annealing, stress relief annealing, and hardening.

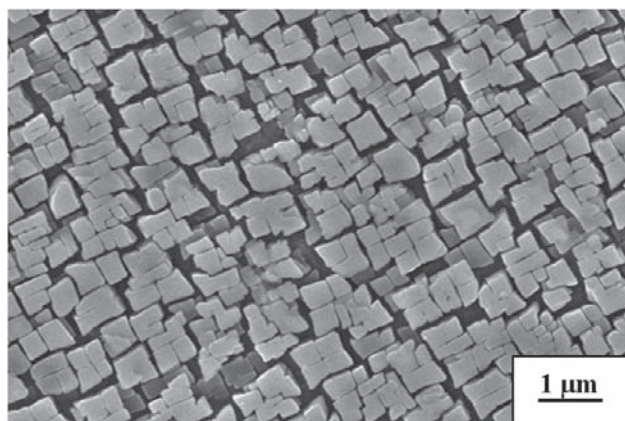


Figure 1 Phase  $\gamma'$  among dendrites

Table 1 Chemical composition of the alloy /wt. %

C	Cr	Mo	Al	Ti	Fe	W
0,16	8,60	0,80	5,60	1,00	0,20	10,00
Ta	Zr	Co	Hf	B	Ni	
3,00	0,06	10,00	1,50	0,02	rest	

Hardening heat resistant alloys is a demanding heat treatment. Optimal combination of quantity, shape, size, and elimination of the hardening phase in the volume of the alloy is achieved by a heat treatment consisting of solution annealing, cooling, and subsequent artificial aging. The solution annealing temperature must be high enough for complete dissolution of the  $\gamma'$  phase.

Depending on the chemical composition of the alloy, it ranges between 1 080 °C and 1 220 °C. The heating from 2 to 12 hours is performed in vacuum furnaces or furnaces with a protective atmosphere to prevent the depletion of the component surface by alloying elements. Cooling from the solution annealing temperature is generally carried out in air. Quenching in water leads to cracking, particularly in high-alloyed alloys with low ductility.

Aging is carried out at heating to a temperature higher than the temperature of the component feature, in the extreme case to the same temperature. Aging temperatures range between 700 °C and 950 °C. During the aging, precipitates of the  $\gamma'$  phase and carbides are eliminated from the supersaturated solid solution. Some nickel alloys are subjected to stepped aging, which achieves the best possible elimination of the  $\gamma'$  phase, accompanied by higher heat resistance. The result is usually bimodal distribution of the concerned phase.

Microstructural analysis of the nickel superalloy was performed on the samples in initial state and after the mentioned modes of heat treatment after chemical etching. Microstructure was analysed with use of light metallographic microscope Olympus GX51. Electron-microscopic analysis using a Quanta FEG 450 scanning electron microscope with a TRIDENT – APEX – 4 X-ray micro-analyser.

It is very convenient to use for temperature measurements infrared technique because it offer contactless temperature measurements. Schema of the measuring system is in the Figure 2 [8].

The measured samples must have an area of about (10 x 10 x 2) mm<sup>3</sup> and must be finely ground. Matt

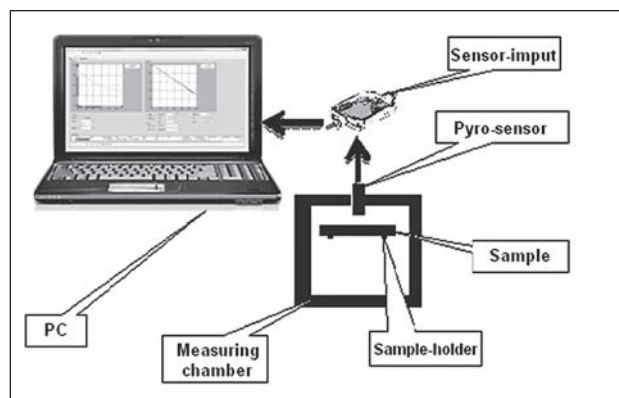


Figure 2 The sketch of thermal tester [8]

black spray-paint is applied on all sides of the samples in order to ensure they have the same emissivity.

Image analysis has been performed by system *Image-Pro Plus*. It has been evaluated the free space between blocks of  $\gamma'$ , (marked as samples number in the Table 2) and number of samples per unit area at the same magnification of a microscope (10 000 x).

## RESULTS AND DISCUSSION

On the base of these assumptions we can start discussion concerning the influence of  $\gamma'$  dissolving on thermal conductivity. In the Table 2 we can see trends of density ( $\rho$ ), thermal conductivity ( $k$ ) and structural factor caused by dissolving process of  $\gamma'$  phase. We can see that for samples 1, 2 and 3 the values of thermal conductivity decrease for dissolving temperatures 900 °C and 1 040 °C. For the same samples decreases also the density as well as the number of samples (free space between  $\gamma'$  blocks). On the other hand with increasing dissolving temperature in the same temperature interval increases number of samples per area (observed free space per the same evaluated area). It is probably caused by increase of free space among particles (blocks) of  $\gamma'$  phase. In this phase of annealing the structure become more rough and less compact which is reflected in worse thermal transport. When the dissolving temperature increase to the 1200 °C and 1240 °C, the structure of  $\gamma'$  phase became fine which is reflected by increase of density, thermal conductivity, increase number of samples and subsequent decrease of number of samples per area in image analyze.

Table 2 Experimental data

sample	k/ W/(m·K)	samples/ number	samples per area/ %	$\rho$ / kg / m <sup>3</sup>
1	81,70	1 185	7	8 226,0
2	66,90	1 022	7	7 309,5
3	51,45	564	13	6 636,6
4	71,80	2 218	8	7 857,4
5	73,05	3 085	7	8 166,4

After application of dissolving annealing in the mode of 900 °C/2 h/water (sample 2) no important changes occurred in microstructure. During the modes working with higher temperatures of dissolving annealing more significant changes in composition, distribution and morphology of minority phases take place. After dissolving annealing in the mode of 1 040 °C/2 h/water (sample 4) the hafnium carbides begin to precipitate in the area of eutectics, between large particles of  $\gamma'$ . Minority phase rich in Mo, W, Cr, probably carbides  $M_{23}C_6$ , is also present. At the same time particles in interdendritic spaces begin to coagulate and to become coarser.

In the point of view of thermal conductivity the presence of carbides contribute probably only slightly to changes of thermal conductivity. Different types of carbides are created in whole temperature interval under investigation also in this case when the temperature conductivity decreases.

## CONCLUSIONS

It may be stated that changes in structure of the investigated alloy took place from the temperature of dissolving annealing of 1 040 °C. These processes are reflected on changing values of thermal conductivity.

It has been shown the dominant role of  $\gamma'$  phase on behaviour of heat flux in such material. Sample density and structural parameters under investigation support this assumption. Increasing temperature of dissolving annealing brought gradually precipitation and increasing frequency of occurrence of hafnium carbides, dissolution of eutectics, change of shape and size of particles  $\gamma'$  both in interdendritic spaces and also inside dendrites. Bimodal distribution was gradually suppressed.

The presented analysis may serve for completion of data and expansion of knowledge about the investigated alloy for industrial practice.

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## REFERENCES

- [1] H. He, Z. Liu, W. Wang, C. Zhou. Microstructure and hot corrosion behaviour of Co-Si modified aluminide coating on nickel based superalloys, *Corrosion Science*, 100 (2015), 466-473.
- [2] L. A. Reyes, P. Paramo, A. S. Zamarripa, M. Garza, M. P. Guerrero Mata. Grain size modeling of a Ni-base superalloy using cellular automata algorithm, *Materials & Design*, 83 (2015), 301-307.
- [3] Y. C. Lin, X. Y. Wu, X. M. Chen, J. Chen, D. X. Wen, J. L. Zhang, L. T. Li. EBSD study of a hot deformed nickel-based superalloy. *Journal of alloys and compounds*, 640 (2015), 101-113.
- [4] I. S. Kim, B. G. Choi, J. E. Jung, J. Do, C. Y. Jo. Effect of microstructural characteristics on the low cycle fatigue behaviors of cast Ni-base superalloys. *Materials Characterization*, 106 (2015), 375-381.
- [5] A. Hernas, Z. Jonšta, M. Tvrđý, L. Čížek, J. Purmenský. *Žárupevné oceli a slitiny [Refractory Steels and Alloys]*. 2<sup>nd</sup> ed. ES VŠB-TU Ostrava, 2009, 392 p. ISBN 978-80-248-2187-0.
- [6] E. I. Galindo-Nava, L. D. Connor, C. M. F. Rae. On the prediction of the yield stress of unimodal and multimodal gamma nickel-base superalloys, *Acta Materialia*, 98 (2015), 377-390.
- [7] Y. Zhang, B. Hu. Function of microelement Hf in powder metallurgy nickel-based superalloys, *Acta Metallurgica Sinica*, 51 (2015) 8, 967-975.
- [8] P. Košťál, I. Špička, Z. Jančíková, J. Valíček, M. Harničárová, J. Hlinka. On experimental thermal analysis of solid materials, *Measurement Science Review*, 14 (2014) 6, 317-322.

**Note:** The translator responsible for English language is Ing. Boris Škandera, Ostrava, Czech Republic