

STRUCTURAL PHASE ANALYSIS OF NICKEL SUPER-ALLOY INCONEL 713LC

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STRUKTURNĚ FÁZOVÁ ANALÝZA NIKLOVÉ SUPERSLITINY INCONEL 713LC

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Abstrakt

V leteckém průmyslu se pro lopatky turbín tryskových motorů často používá niklových superslitin. Je to proto, že na materiál je kladena řada extrémních požadavků, jako např. žárupevnost za vysokých teplot, odolnost vůči únavovému poškození, odolnost vůči agresivnímu působení spalin aj. Dlouhodobá životnost a spolehlivost materiálu je přímo spojena s jeho mikrostrukturou, resp. s její stálostí při dlouhodobé exploataci. Použité materiály bývají komplexně legovány a jsou ze strukturního hlediska značně komplikované [1, 2]. Jedním z materiálů, který se používá pro uvedené aplikace, je niklová superslitina INCONEL 713LC komplexně legovaná Cr-Al-Mo-Ti-Nb-Zr používaná ve formě odlitků. V dané slitině se uplatňuje několik zpevňujících mechanismů, hlavním je precipitační zpevnění koherentními precipitáty intermetalické fáze Ni_3Al , resp. $\text{Ni}_3(\text{Al},\text{Ti})$ [3]. Niklová superslitina má ve stavu po odlití heterogenní strukturu s výraznými dendritickými segregacemi. V oblastech segregací byly nalezeny částice karbidů, resp. karbonitridů niobu a titanu typu $(\text{Nb},\text{Ti})(\text{C},\text{N})$, dále rozměrné částice intermetalické fáze typu Ni_3Al a konečně vícefázové útvary s vyšším obsahem Mo, Nb, ale zejména Zr, které se nepodařilo přesně identifikovat. Předložený příspěvek je zaměřen na studium struktury odlitků z niklové superslitiny INCONEL 713LC po různých variantách tepelného zpracování. Laboratorní žhání při teplotě 1240°C ukázalo, že v materiálu byly, pravděpodobně již ve výchozím stavu, přítomny jemné řetízky karbidických částic na hranicích zrn. Samotné vysokoteplotní žhání nevedlo k precipitaci zpevňující intermetalické fáze γ' .

Abstract

Aircraft industry often uses nickel super-alloys for blades of jet engine turbines. 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 combustion products, etc. Long-term service life and reliability of material is directly related to its microstructure, i.e. to its stability at long-term exploitation. Used materials are usually alloyed in a complex manner and their structure is very complicated [1, 2]. One of materials used for these applications is cast, complex alloyed nickel super-alloy INCONEL 713LC on the basis of Cr-Al-Mo-Ti-Nb-Zr. Several strengthening mechanisms take effect in this alloy, the main mechanism is precipitation strengthening by coherent precipitates of inter-metallic phase Ni_3Al , or $\text{Ni}_3(\text{Al}, \text{Ti})$ [3]. Nickel super-alloy has in as cast state heterogenous structure with distinct dendritic segregations. In the zones of segregations there were found particles of carbides or niobium and titanium carbo-nitrides of the type $(\text{Nb}, \text{Ti}) (\text{C}, \text{N})$, moreover large particles of inter-metallic phase of the type Ni_3Al , and finally multiphase formations with higher

contents of Mo, Nb, but particularly of Zr, the reason of which we were unable to identify precisely. This paper is focused on investigation of structure of castings made of nickel super-alloy INCONEL 713LC after various variants of heat treatment. Laboratory annealing at the temperature of 1240 °C showed that fine strings of carbidic particles were present in material at the grain boundaries, probably already in initial state. Heat treatment 1240 °C/2 hours/furnace to 940 °C/air already triggered intensive precipitation of the phase γ' .

Keywords: structural phase analysis, heat treatment, inter-metallic phase γ' , nickel super alloy.

1. Experimental procedure

We have used for experimental investigation castings made of commercially produced nickel super-alloy INCONEL 713 LC. Chemical composition of the used melt is given in the Table 1.

Table 1 Chemical composition of nickel super-alloy INCONEL 713LC (mass %)

C	Cr	Al	Mo	Ti	Nb	Zr	Ni
0.05	9.3	6.5	4.7	0.8	2.1	0.1	rest

Material was investigated in initial as cast state, and also after three modes of laboratory heat treatment:

- 1240 °C/2 hours/water,
- 1240 °C/2 hours/air,
- 1240 °C/2 hours/furnace to 940 °C/air.

Material was after heat treatment subjected to detailed structural analysis with use of methods of light microscopy, or scanning electron microscopy on microscopes OLYMPUS IX71 and JEOL JSM 50A. Material microstructure was developed by chemical etching in solution of cupric chloride, hydrochloric acid and distilled water. Local analysis of chemical composition was made by electron micro-analyser JCSA 733 with use of energy dispersive analyser EDAX.

2. Results and discussion

2.1 Initial as cast state

Material's structure in initial state was somewhat heterogenous and it is documented in Fig. 1 a, b, c. Dendritic structure of the alloy is visible very well in Fig. 1 a, b. In the areas of dendritic segregations we observed three different types of particles, which were afterwards identified by method of local analysis of chemical composition. Example of particles is shown also in Fig. 2 in reflection of secondary electrons. Individual types of particles are marked A, B, C – identically in figures 1 c and 2.

Particles of the type A were identified by method of local micro-analysis as carbides, or niobium and titanium carbo-nitrides of the type (Nb, Ti) (C, N). Niobium prevailed in these particles, relation of niobium and titanium was approx. 8:1. These particles formed locally distinct eutectics, they were locally segregated in discrete rows in inter-dendritic spaces.

We have established in case of the particles marked B, that they are probably inter-metallic particles of the type Ni_3Al , which were formed already during solidification of the casting. Typical EDX spectrum obtained at local micro-analysis of particles of the type B is shown in Fig. 3. Particles were present in samples only locally.

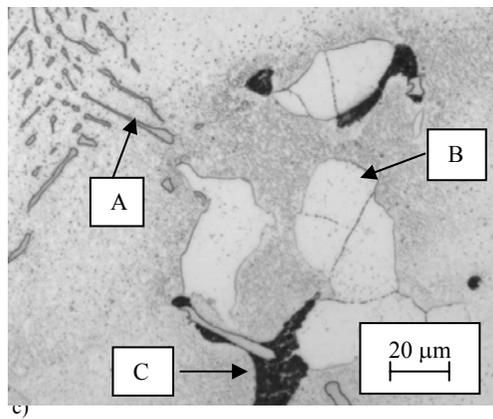
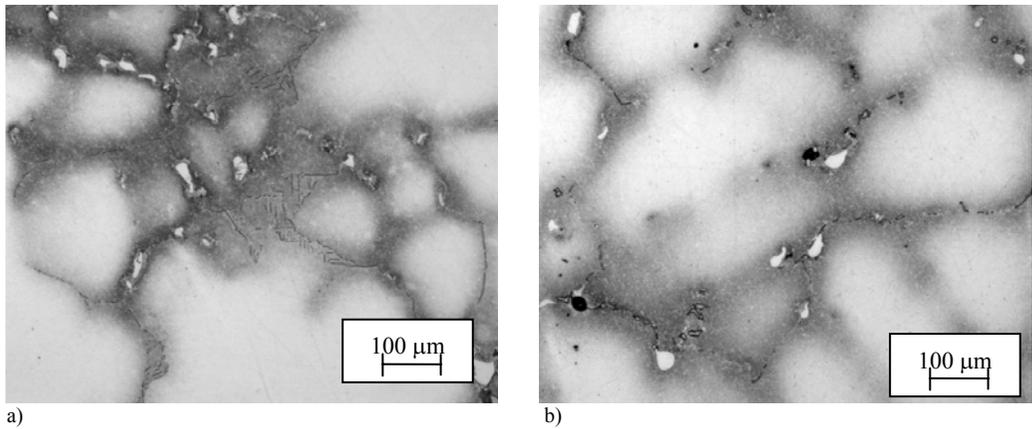


Fig.1 a, b, c Microstructure of the alloy in initial as cast state

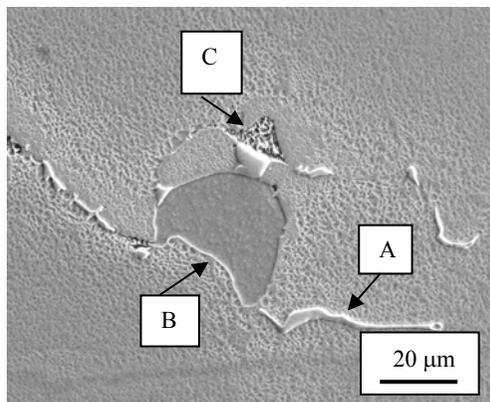


Fig.2 Microstructure of the alloy in initial as cast state with various types of particles

Representation of formations marked as C, with use of method light microscopy as well as scanning electron microscopy, indicates, that this is not a single phase but product of

eutectic, or eutectoid reaction. Dimensions of individual particles in these formations, however, did not make it possible to make their reliable identification. Average EDX spectrum obtained at local micro-analysis of formations marked as C is shown in Fig. 4 which shows also average contents of Zr among these values, due to the fact that its average contents in investigated alloy is only 0.1 weight %. It can be deduced from the morphology of particles C that they were appeared only after formation of the particles of the type A and B, i.e. niobium and titanium carbo-nitrides, or of greater formations of inter-metallic phase of the type Ni_3Al . Particles of the type C were also, similarly as particles of the type B, present only locally, i.e. that their segregation was uneven.

Apart from coarser particles discussed above, which were present in the areas of dendritic segregations, we have observed in the matrix also numerous fine precipitates. In this case they were unequivocally strengthening precipitates of the phase γ .

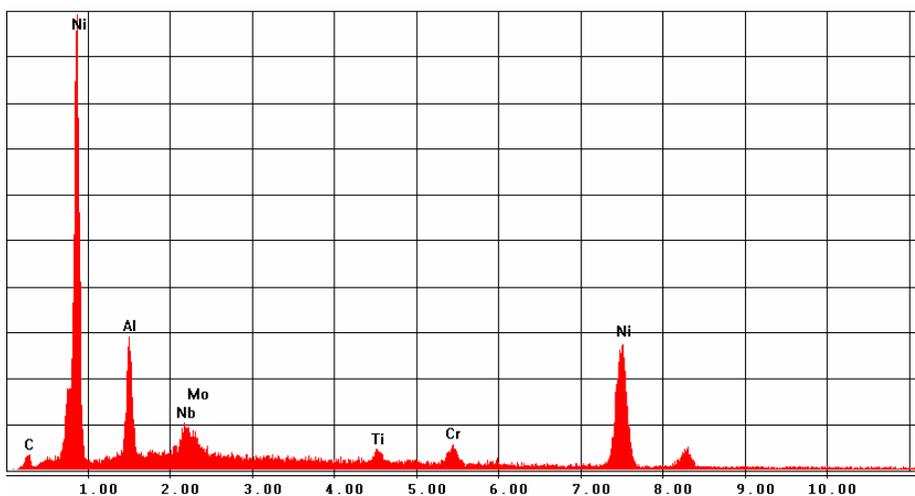


Fig.3 Typical EDX spectrum of particles of the type B (inter-metallic phase of the type Ni_3Al)

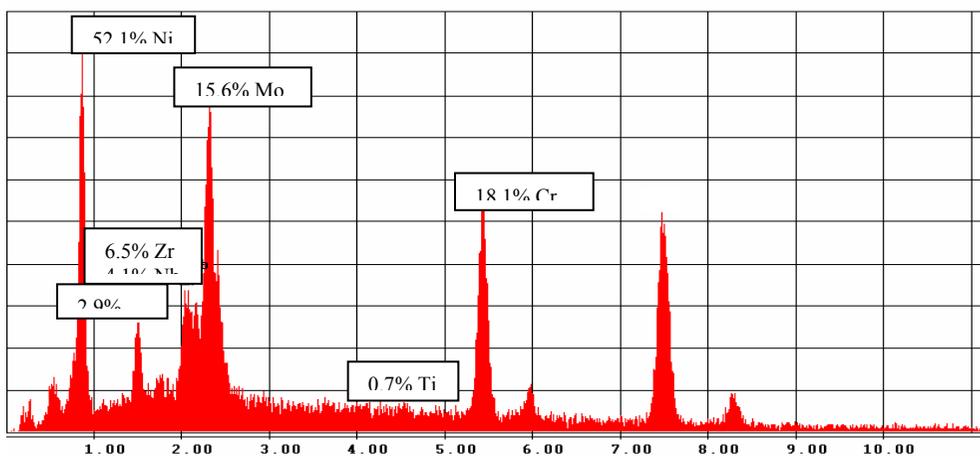


Fig.4 Typical EDX spectrum of particles of the type C with presentation of average values of the contents of analysed elements

We have not observed in initial state strings of fine particles of carbides on boundaries of casting grains. These precipitates are dangerous, since they can lead to premature development of defects and reduce significantly reliability and shorten thus service life of turbine blades [4]. It is, however, necessary to take into account that boundaries of casting grains coincided largely with areas of dendritic segregations and they therefore appeared as dark areas after etching. Identification of fine carbidic precipitates would be rather problematic in these areas.

2.2 State after heat treatment by regime 1240 °C/2 hours/water

Material's microstructure after this heat treatment is documented by light microscopy method in Fig. 5 a, b.

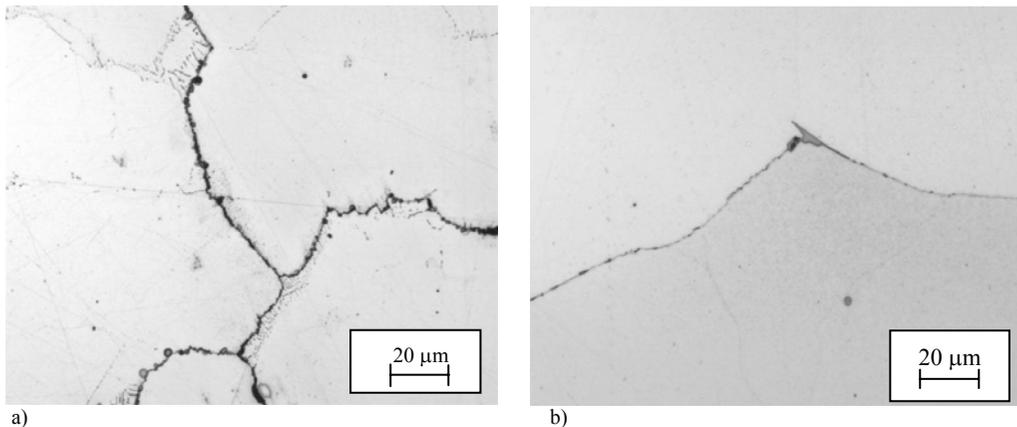


Fig.5 Microstructure of the alloy after heat treatment 1240 °C/2 hours/water

It is obvious from photos that after heat treatment dendritic structure of the alloy is almost not etched anymore (Fig. 5 a). It means therefore, that there occurred at least partial homogenisation of its chemical composition. We have, however, observed in material comparatively frequent inter-crystalline cracks (fig. 5 a). We have found after detailed analysis that very fine precipitates covered at part of grain boundaries, at the boundary discernible by light microscopy method (Fig. 5 b). These are probably very finely dispersive carbides, or niobium and titanium carbo-nitrides of the type (Nb, Ti) (C, N). Since it is highly improbable that these particles were formed during the heat treatment, we can assume that they were present in the alloy already in its initial state, but we were unable to identify them. The works [5, 6] state, that particles of this type dissolve at temperatures of 1200-1260 °C. In the given case, nevertheless, dwell of 2 hours at the temperature of 1240 °C was probably insufficient for full dissolving of these precipitates. Weakening of grain boundaries by present particles then resulted in formation of cracks at rapid cooling (water quenching) due to high local stresses in material. We have not observed in matrix after this heat treatment segregation of fine particles of inter-metallic phase γ' .

2.3 State after heat treatment by regime 1240 °C/2 hours/air

Microstructure of the alloy was after this heat treatment identical with the previous state. The only difference was, that after cooling on air no cracks were observed on grain

boundaries. This supports a hypothesis that cracks in the previous case originated due to stress at rapid cooling in water as a consequence of local weakening of grain boundaries by fine precipitates of the type (Nb, Ti) (C, N).

2.4 State after heat treatment by regime 1240 °C/2 hours/furnace to 940 °C/air

In this case structure of the alloy was already different. There occurred a real etching of structure as a consequence of precipitation of the strengthening inter-metallic phase γ' . No fine precipitates were observed at the grain boundaries. Their presence cannot, however, be completely excluded. In this case as well it is possible that character of structure prevents their visibility. Microstructure of the alloy after heat treatment is shown in Fig. 6 a, b.

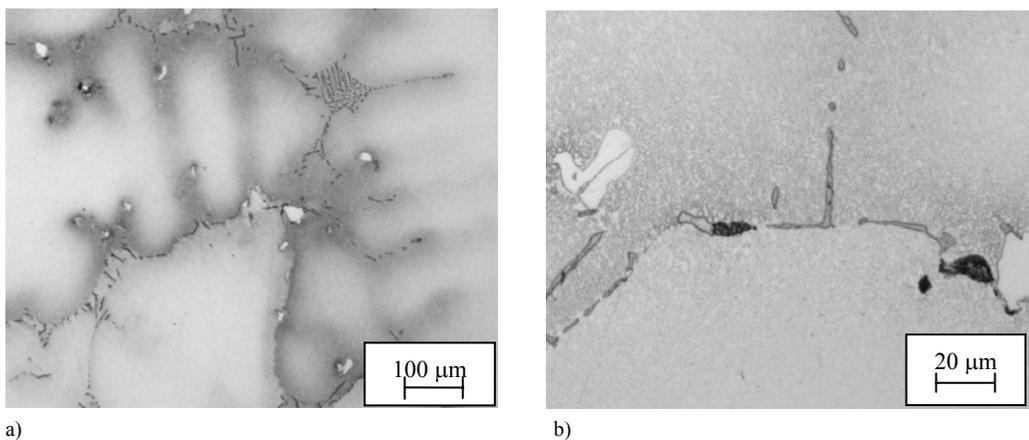


Fig.6 Microstructure of the alloy after heat treatment 1240 °C/2 hours/furnace to 940 °C/air

3. Conclusions

Nickel super-alloy has in as cast state heterogenous structure with distinct dendritic segregations. In the zones of segregations there were found particles of carbides or niobium and titanium carbo-nitrides of the type (Nb, Ti) (C, N), moreover large particles of inter-metallic phase of the type Ni_3Al , and finally multiphase formations with higher contents of Mo, Nb, but particularly of Zr, the reason of which we were unable to identify precisely. Completed laboratory heat treatment changed distinctly material structure. Laboratory annealing at the temperature of 1240 °C showed that fine strings of carbidic particles were present in material at the grain boundaries, probably already in initial state. They afterwards caused during cooling by water development of inter-crystalline cracks. High-temperature annealing as such did not lead to precipitation of the strengthening inter-metallic phase γ' . Heat treatment 1240 °C/2 hours/furnace to 940 °C/air already triggered intensive precipitation of the phase γ' . We have not observed precipitates at the grain boundaries, it is, however, to exclude their presence. On the basis of obtained results it is possible to recommend a heat treatment, which would consist of heating and dwell at the temperature exceeding 1240 °C (min. 1260 °C), so that precipitates at the grain boundaries dissolve completely, with subsequent slow cooling down to the temperature of approx. 940-950 °C, so that there occurs intensive intra-granular precipitation of inter-metallic phase γ' .

Acknowledgements

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