

Nickel super alloy INCONEL 713LC – structural characteristics after heat treatment

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Materials

ABSTRACT

Purpose: Nickel super alloy's products are mainly using for construction parts of jet engines, gas turbines and turbo-blowers.

Design/methodology/approach: Super alloy was commercially produced and was investigated by using the light microscopy (OLYMPUS IX 71) and local chemical microanalysis and by the scanning electron microscopy (JEOL JSM 50A)

Findings: We found a mode of optimum heat treatment. On the basis of obtained results it is possible to recommend a following regime of heat treatment: 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 intermetallic phase γ' .

Research limitations/implications: The experiment was limited by occurrence a void in cast alloys.

Practical implications: Nickel super alloy's products are mainly using for construction parts of jet engines, gas turbines and turbo-blowers.

Originality/value: Mode of optimum heat treatment was proposed. On the basis of obtained results it is possible to recommend the most suitable heat treatment, which produce intensive intra-granular precipitation of intermetallic phase γ' . It was received a new know-how in this field.

Keywords: Metallic alloys; Nickel super-alloy; Structural phase analysis; Intermetallic phase γ' ; Heat treatment

1. Introduction

Nickel super alloy's products are mainly using for construction parts of jet turbines, gas turbines and turbo-blowers. It is so because this material must satisfy numerous extreme requirements, for example heat resistance at high temperatures, resistance to aggressive effect of combustion products, resistance to fatigue damage and so on. [1 - 5]. 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 [6 - 10]. One of materials used for these important applications is in cast, complex alloyed nickel super-alloy INCONEL 713LC alloying by Cr-Al-Mo-Ti-Nb-Zr. Several strengthening mechanisms take effect in this alloy, the main mechanism is precipitation strengthening by coherent precipitates

of intermetallic phase Ni_3Al , or $\text{Ni}_3(\text{Al}, \text{Ti})$ [11 - 16]. The paper summarized on investigation of structure of castings made of nickel super-alloy INCONEL 713LC after various variants of heat treatment.

2. Used material and experimental technique

Nickel super alloy INCONEL 713LC was commercially produced. Chemical composition of the used melt is shown 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 (HT). First of all was $1240^\circ\text{C} / 2 \text{ hours} / \text{water}$, secondly $1240^\circ\text{C} / 2 \text{ hours} / \text{air}$ and the last regime was $1240^\circ\text{C} / 2 \text{ hours} / \text{furnace}$ to $940^\circ\text{C} / \text{air}$.

After heat treatment a detailed structural analysis of material was carried out. For light microscopy (LM) we used microscope OLYMPUS IX71 and scanning electron microscopy (SEM) was carried out on device JEOL JSM 50A. Microstructure of material 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.

3. Results of tests and discussion

3.1. Initial as cast state

Structure of material in initial state was somewhat heterogenous and it is documented in Fig. 1a, b, c. Dendritic structure of the alloy is visible very well in Fig. 1a, b. We observed three different types of particles in the areas of dendritic segregations, 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 and are demonstrate 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 carbonitrides of the type $(\text{Nb}, \text{Ti}) (\text{C}, \text{N})$. 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 intermetallic 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.

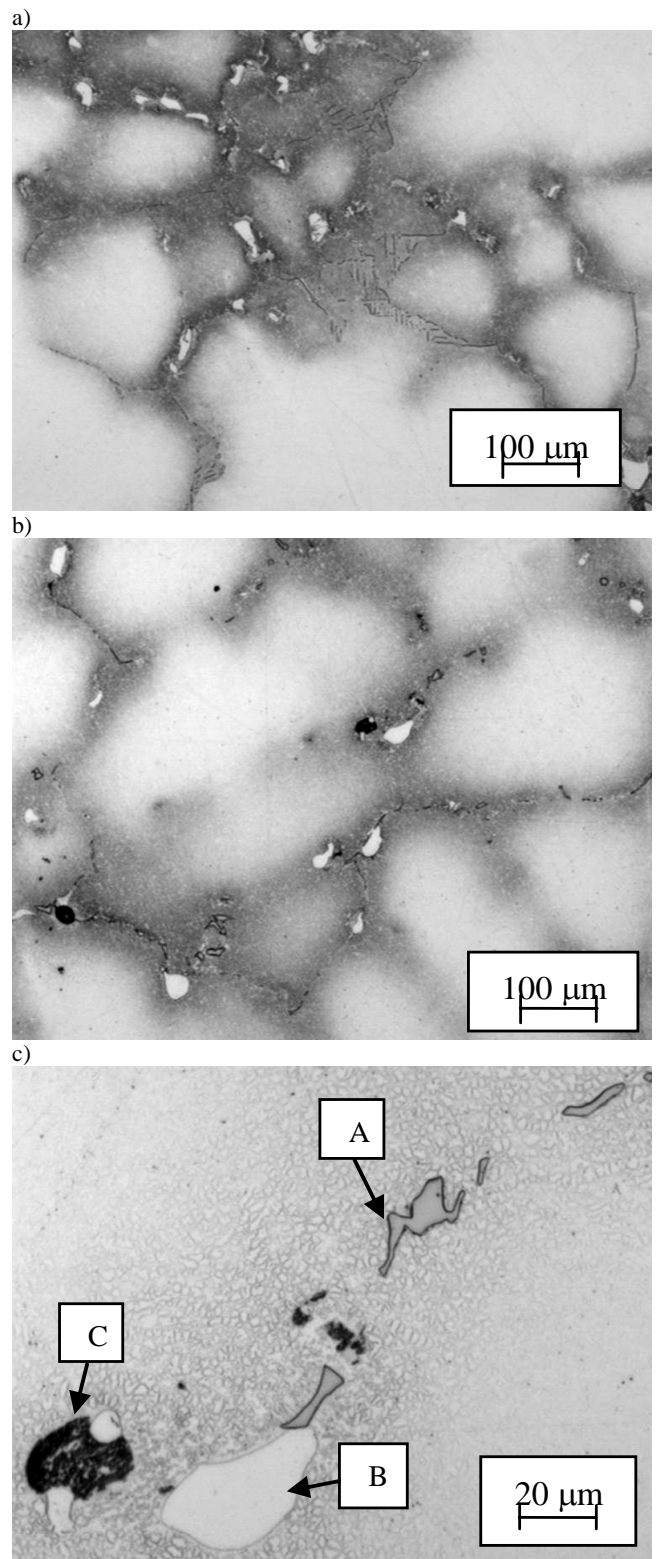


Fig. 1. Microstructure of the alloy in initial as cast state (LM)

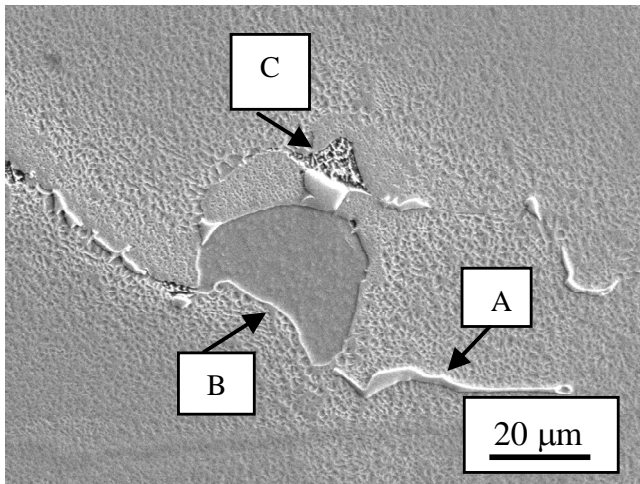


Fig. 2. Microstructure of the alloy in initial as cast state with various types of particles (SEM)

Representation of formations marked as C, with use of method LM as well as SEM, indicates, that this is not a single phase, but products of eutectic, or eutectoid reaction. Dimensions of individual particles in these formations, however, did not make it possible to make their reliable identification. Average chemical composition obtained at local micro-analysis of formations marked as C is shown in Fig. 4. We would like to draw attention to comparatively high contents of Zr among these values, due to the fact that its average content in investigated alloy is only 0.1 mass %. 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 particles 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 distribution 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' .

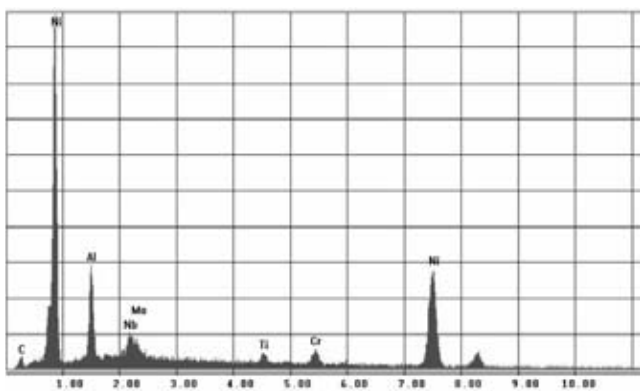


Fig. 3. Typical EDX spectrum of particles of the type B (intermetallic phase of the type Ni_3Al)

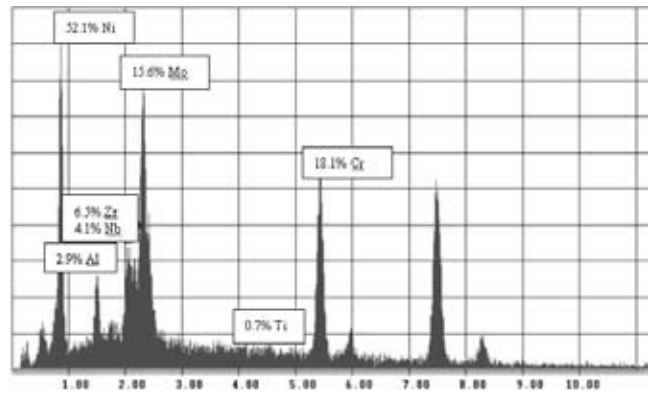


Fig. 4. Typical EDX spectrum of particles of the type C with presentation of average values of the contents of analysed elements

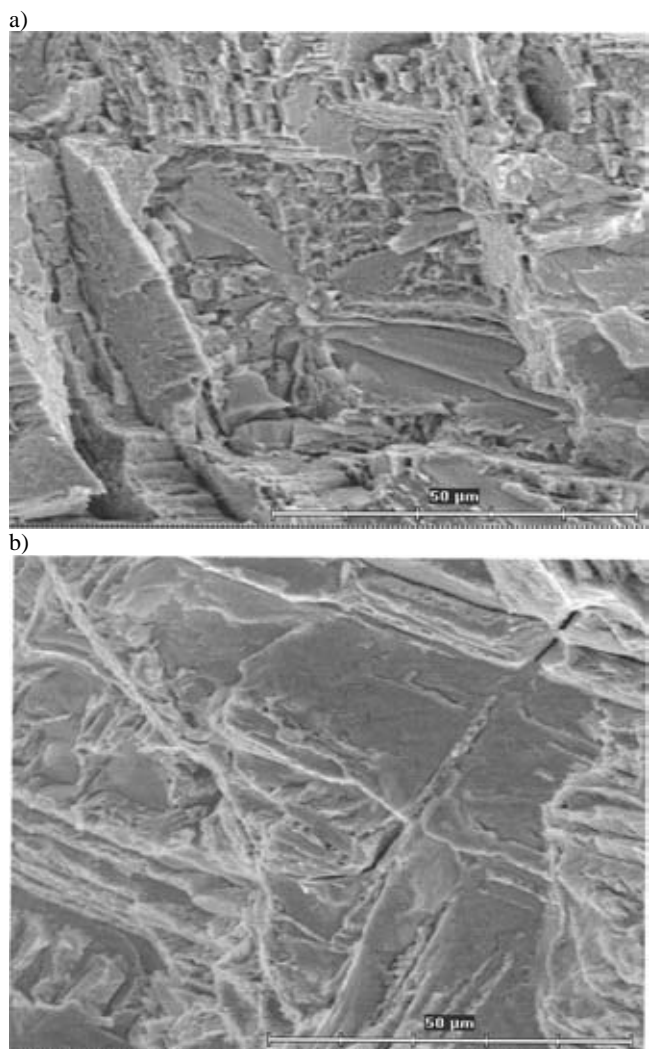


Fig. 5. Fracture areas of the alloy in initial as cast state (SEM)

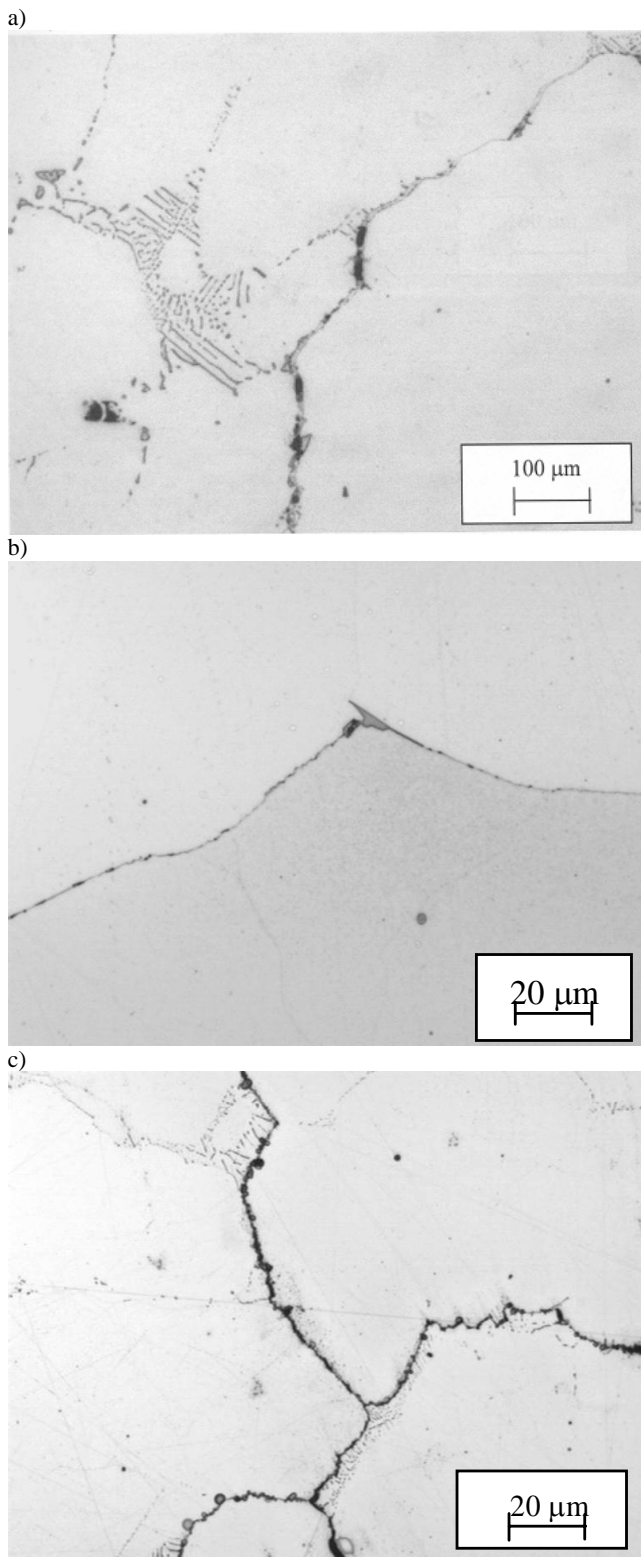


Fig. 6. Microstructures of the alloy after HT 1240 °C / 2 hours / water (LM)

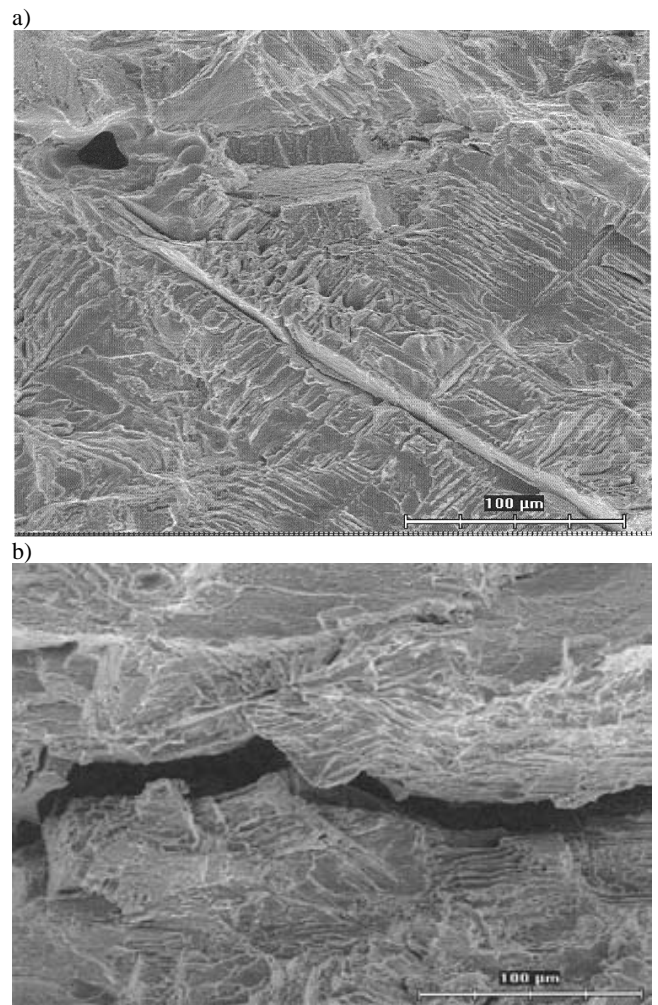


Fig. 7. Results of fracture analysis of the alloy after HT 1240 °C / 2 hours / water (SEM)

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 [17 - 20]. 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. Material was also investigated by the SEM on samples, which were taken from bodies of specimens after tensile tests. Fracture areas are shown in fig. 5 a, b. On fracture areas were predominantly brittle facets of transcrystalline character.

3.2. State after HT 1240 °C / 2 hours / water

Microstructure of material after this HT is documented by LM method in fig. 6 a, b, c.

It is obvious from photos that after heat treatment dendritic structure of the alloy is almost not etched anymore (fig. 6c). It means therefore, that there occurred at least partial homogenisation of its chemical composition.

We have, however, observed in material comparatively frequent intercrystalline cracks (fig. 6c). We have found after detailed analysis that very fine precipitates covered at part of grain boundaries, at the boundary discernible by LM method (fig. 6b). These are probably very finely dispersive carbides, or niobium and titanium carbonitrides 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 [1, 5] 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 HT segregation of fine particles of inter-metallic phase γ' . Results of fracture analysis are given in fig. 7a, b.

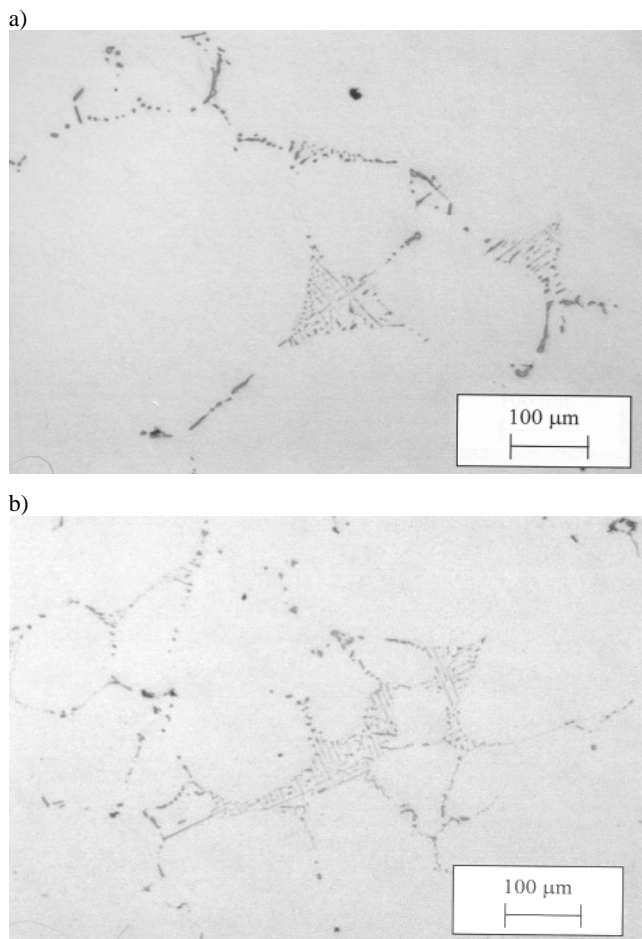


Fig. 8. Microstructures of the alloy after HT 1240 °C / 2 hours / air (LM)

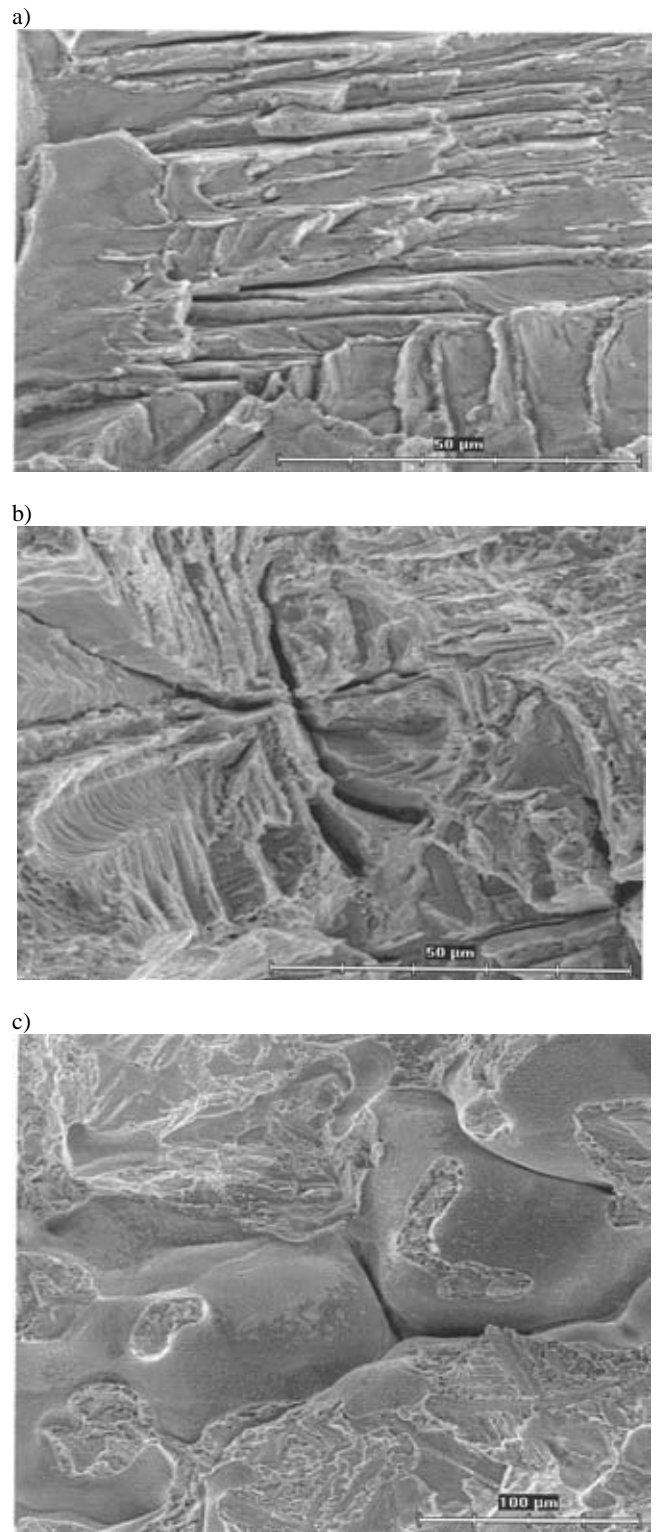


Fig. 9. Fracture areas of the alloy after HT 1240 °C / 2 hours / air (SEM)

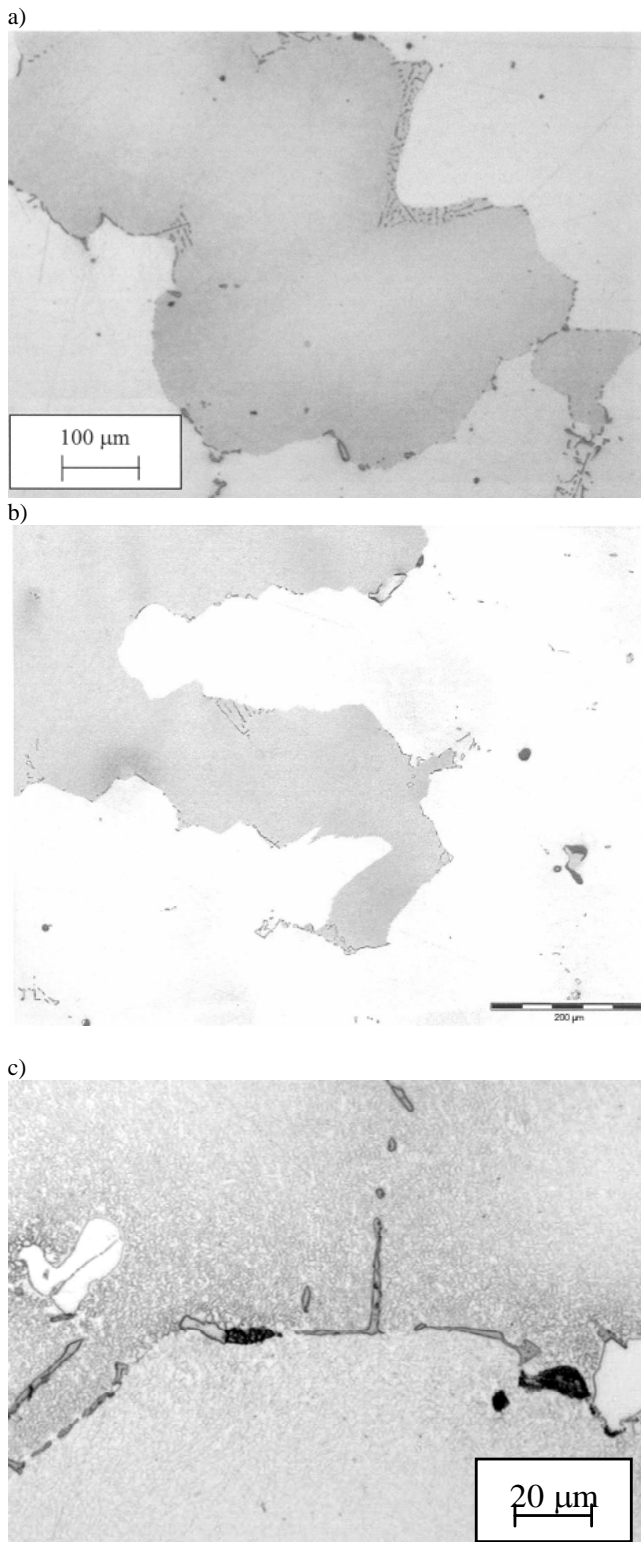


Fig. 10. Microstructure of the alloy after HT 1240 °C / 2 hours / furnace to 940 °C / air (LM)

3.3. State after HT 1240 °C / 2 hours / air

Microstructure of the alloy after this HT was identical with the previous state and are shown in fig. 8a, b. 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).

Material was also investigated by the SEM. Fracture areas are shown in fig. 9a, b, c.

3.4. State after HT 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 HT is shown in fig. 10a, b, c. Fracture areas of the alloy after HT 1240 °C / 2 hours / furnace to 940 °C / air. are in fig. 11a, b.

4. Conclusions

Results shown that nickel super alloy has in as cast state heterogenous structure with distinct dendritic segregations. It was obvious from analyses, in the zones of segregations there were found particles of carbides or niobium and titanium carbonitrides of the type (Nb, Ti) (C, N), moreover large particles of intermetallic 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. Structure of investigated material was after laboratory heat treatment evidently changed. Laboratory annealing at the temperature of 1240 °C showed that fine strings of carbide particles were present in material at the grain boundaries, probably already in initial state. They afterwards caused during cooling by water development of intercrystalline cracks. High-temperature annealing as such did not lead to precipitation of the strengthening intermetallic 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. Obtained results from analyses led to recommend an optimal regime of heat treatment: 1240 °C/2 hours/furnace to 940 °C/air. For the first time this regime caused that precipitates at the grain boundaries dissolved completely and secondly it was observed intensive intra-granular precipitation of strengthening phase γ' .

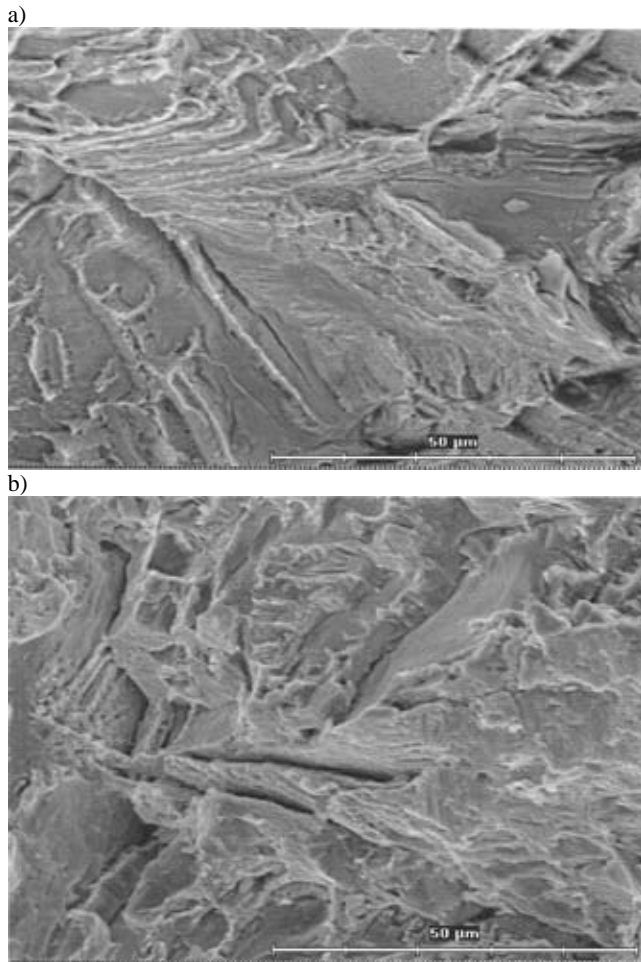


Fig. 11. Fracture areas of the alloy after HT 1240 °C / 2 hours / furnace to 940 °C / air (SEM)

Acknowledgements

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