Electron Microscopy Researches on the Microstructure of Inconel 718 Alloy

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Identification and characterization of basic austenitic matrix, carbides, and hardening precipitates present in superalloys are crucial for establishing and controlling the heat treatments to which the material must be subject in order to achieve the required technological parameters: high mechanical properties, resistance to creep, fatique and corrosion. The microstructure of Inconel 718 nickel-iron based superalloy, used for execution of anchoring heads in Cernavoda nuclear power station units, is characterized and analyzed in this paper. Forged Inconel 718 superalloy was subject to the following heat treatment: solution annealing treatment at 1000°C, air cooling, and two steps ageing treatment (heating at 771°C for 8 h, cooling in furnace up to 660°C, 10 h maintained at that temperature, air cooling). Several calibrations of the heat treatment furnace were carried out to meet the required heat treatment regime (very restrictive ± 1°C). The microstructure was investigated by optical and electron microscopy using JEOL 100 CX / ASID 2D equipment and by energy-dispersive X-ray spectroscopy (EDX). Two-steps replicas were used for the identification and characterization of the phases present in the austenitic matrix. The ratio of the alloy phases is affected by small variations in the applied heat treatment. The purpose of the study was to investigate the characteristic phases present in the Inconel 718 superalloy, especially identification and analysis of the $\gamma''(Ni_3Nb)$ hardening precipitates morphology.

Keywords: γ', γ'' Ni_3Nb hardening precipitates, Fe-Ni superalloys, heat treatments, Inconel 718 alloy

Superalloys have been developed having nickel or cobalt as basis, and followed lately by the forged Fe-Ni based superalloys [1]. Nickel-based alloys are metallic materials of high technical importance due to their special properties: excellent characteristics both at elevated- and low-temperatures, refractory and corrosion resistance, outstanding magnetic properties (variable or constant magnetic permeability), wear resistance and corresponding anti-friction properties, practically zero expansion coefficient in the 0...100°C temperature range, very close to that of platinum and glass, high electrical resistance, etc.

Among the refractory nickel-based alloys with high resistance at elevated temperatures can be mentioned sumonic, hasteloy, caronel, illium, inconel, incoloy, udimet, termaloy, stervac, piromet, waspalloy.

Inconel alloys are sufficiently resistant to the action of chlorine, fluorine, and solutions containing ions of these elements, and are not vulnerable to water, vapour, and carbon dioxide or ammonia, nitrogen and hydrogen mixture, showing better resistance to progressive oxidation up to 1100°C and in the oxidizing-sulphide atmosphere up to 850°C. Summing these properties the Inconel alloys are intended for manufacturing of steam and gas turbines, and parts for aerospace and nuclear industries [2].

The chemical compositions of some nickel-based superalloys from Inconel group are given in table 1.

Characteristic phases in Inconel 718 superalloy

Austenitic matrix

The matrix is a hardened CFC type solid solution, the hardening effect being proportional to the difference between the atomic size of the matrix (Ni) and atomic size of the dissolved elements.

The dissolved elements in the solid solution affect the hardening in the following order: Cr, Mo, and W (table 2).

Table 1
CHEMICAL COMPOSITION OF NICKEL-BASED ALLOYS (INCONEL) [3]

ALLOY	CHEMICAL COMPOSITION, WEIGHT %													
	Ni	Cr	Fe	Mo	Nb	Co	Mn	Cu	Al	Ti	Si	C	S	P
600	72.0	14.0 - 17	6.0 - 10.0				1.0	0.5			0.5	0.15	0.015	
617	44.2 - 56.0	20.0 - 24.0	3.0	8.0 - 10.0		10.0 - 15.0	0.5	0.5	0.8 - 1.5	0.6	0.5	0.15	0.015	0.015
625	58.0	20.0 - 23.0	5.0	8.0 - 10.0	3.15 - 4.15	1.0	0.5	0.2	0.4	0.4	0.5	0.1	0.015	0.015
718	50.0 - 55.0	17.0 - 21.0	Rest	2.8 - 3.3	4.15 - 5.5	1.0	0.35	0.2 - 0.8	0.65 - 1.15	0.3	0.35	0.08	0.015	0.015
X-750	70.0	14.0 - 17.0	5.0 - 9.0		0.7 - 1.2	1.0	1.0	0.5	0.4 - 1.0	2.25 - 2.75	0.5	0.08	0.01	

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Element	Atomic radius (A)	Crystallization system
Ni	1.24	CFC
Cr	1.30	CFC
Mo	1.39	CFC
W	1.41	CFC

Table 2
DISOLVED ELEMENTS IN CFC TYPE SOLID SOLUTION OF INCONEL 718 SUPERALLOY [3]

Element	Effect
Aluminum	*Forms the $Ni_3(Al,Ti)$; delays the γ'- Ni_3Ti phase formation with hexagonal structure;*
Titanium	*Forms the $Ni_3(Al,Ti)$, MC carbides type;*
Niobium	*Forms the Ni_3Nb – phase with orthorhombic structure;*
Carbon	*Forms the MC, M_7C_3; $M_{23}C_6$, M_6C carbides types; stabilizes the CFC matrix;*
Phosphor	*It favours the general precipitation of carbides;*
Nitrogen	*Forms the M(C,N) carbonitrides;*
Chromium	*Plays an important role in increasing the resistance to corrosion, affects the hardening solid solution;*
Nickel	*Stabilizes the CFC matrix; prevents the formation of undesirable phases;*
Tungsten	*Forms the M_6C carbides types;*

Table 3
EFFECT OF ALLOYING ELEMENTS IN THE Fe-Ni ALLOYS

The effect of the major alloying elements in the nickel-based superalloys from the Fe-Ni group is shown in table 3.

Carbides

Most nickel-based alloys contain carbides both at the grain boundary and dispersed throughout the matrix. The most commonly observed types are: MC, M_6C and $M_{23}C_6$; these will be analyzed below.

MC carbides have a block morphology and CFC structure, even though they are formed below the solidification temperature. They have a tendency to decompose with increasing temperature unless the alloy is rich in Nb and Ta, in which case they are stabilized. The carbides type characteristic to this form are TaC, NbC, TiC, and VC, arranged in the decreasing stability order. This sequence is not dictated by temperature, this behaviour being due to the presence of Mo or W that weakens the bonding forces.

Depending on the alloy composition, not only the "M" atoms can substitute each other, but also other less reactive elements can be incorporated into the carbides. For example, in the Rene 95 the MC carbides appear to be very large (2 μm) [3].

$M_{23}C_6$ carbides have a complex cubic structure and tend to form along the grain boundaries, being abundant in alloys with a high Cr content; they tend to be stable at intermediate temperatures (870 to 980°C), depending on the composition.

Similar to the MC-type carbides, they can have a variable composition, depending on the contained metal. For example, when W and Mo are present, the carbide composition can be $Cr_2(Mo,W)_2C_6$. As carbides are located at the grain boundary, they tend to increase the creep resistance by preventing the grain boundary sliding.

Carbides are usual primers for cracking caused by elevated temperatures creep. It is known that the $M_{23}C_6$ carbides and γ'-type precipitates develop at the grain boundary an interpenetrated lattice that is very susceptible to environmental attack, and reduces ductility.

M_6C-type carbides have a complex cubic structure and generally appear as precipitates at the grain boundary (Fig. 1).

The composition varies widely in forms such as $(Ni,Co)_2Mo_3C$ or $(Ni,Ca)_2W_4C$. Stability of these carbides at elevated temperatures may be used to control the grain size resulted from high temperature thermal treatment. These carbides may react with other phases forming carbides or new phases; the reactions are slow, and in some cases continue throughout the alloy lifetime.

The most important reactions for carbides into the Ni-based alloy are:

$$MC + \gamma \rightarrow M_{23}C_6 + \gamma' \quad (1)$$
$$MC + \gamma \rightarrow M_6C + \gamma' \quad (2)$$
$$M_6C + M' \rightarrow M_{23}C_6 + M'' \quad (3)$$

where M' and M" are metals from the matrix [3].

Hardening precipitates

From the structural point of view, the γ'-precipitate represents a Ni_3Al-type phase, with Ni atoms situated in the planes centre and Al atoms (or the elements that can replace Al) in the cube corners.

Usage of the γ'' precipitates is limited for Fe-Ni alloys with high Nb content (~ 5%). Inconel 718 is the most important alloy example in which the γ''-formations have been identified.

The γ'' phase is displayed as plate shape; its volume percentage is substantially higher than the γ' phase. Although both γ''- and γ'-phases will be present in the alloy, the γ''-phase could be the predominant hardening agent if Nb is present in superalloy.

The following transformation between γ' and γ'' is possible [4]:

$$\gamma' \rightarrow Ni_xNb \text{ (body centered tetragonal - } \gamma'') \rightarrow Ni_3Nb \text{ (orthorhombic – } \delta \text{ delta phase)} \quad (4)$$

After a long-time exposure at temperature above 660°C, the γ''-phase, generally unstable, can be converted to the γ'- or to δ-phase (Ni_3Nb).

Although the γ''-phase has not been studied to the same extent as γ'-phase, it can be attributed behavioural considerations by similarity to the γ'-phase. The hardening precipitates γ' (γ'') are particularly important for this type of alloy, because their presence, morphology and distribution in the austenitic based matrix depend on the applied thermal treatment. For this reason, these precipitates are considered structure control phases.

It is possible to group superalloys depending on the phases used to control the structure (table 4).

The working domain (fig. 1) has a lower limit temperature above which the phase is used to provide

Alloy	Control phases	Working temperature (°C)
IN -738	(Ni_3Ti,Al)	816 - 1121
Inconel - 718	(Ni_3Nb)	915 - 995
Waspaloy	(Ni_3Al, Ti)	954

Table 4
CONTROL PHASE IN NI BASED SUPERALLOYS [4]

ALLOY	CHEMICAL COMPOSITION, WEIGHT %													
	Ni	C	Mn	Si	Cr	Co	Mo	W	Nb	Ti	Al	B	Zr	Fe	Cu
Inconel 718	52.86	0.04	0.2	0.2	18.5	...	3.0	...	5.1	0.9	0.5	...	...	18.5	0.2

Table 5
CHEMICAL COMPOSITION OF NICKEL-BASED ALLOYS STUDIED (INCONEL - 718)

structure control, and a upper limit temperature, which if it was exceeded, the structure control phase enters in solution and its effect gets lost.

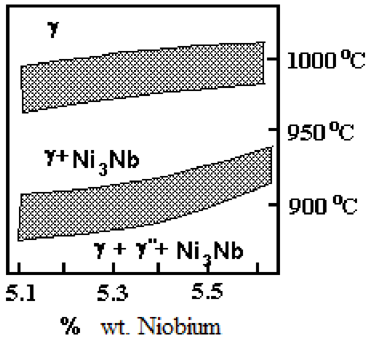


Fig. 1. Control domain for γ' and γ''phases [4].

It can be observed that the control domain for γ' phase and γ'' (Ni_3Nb) respectively, in the case of Inconel 718 superalloy, is contained in a narrow temperatures range, depending on the chemical composition (Niobium content).

Experimental part

Table 5 shows the chemical composition of studied Inconel 718 superalloy.

The applied heat treatments

The heat treatments applied were: solution annealing followed by ageing.

Three thermal treatment cycles were analyzed:

-Sample A - solution annealing to 950°C/1h/air cooling + ageing at 771°C/8h/ furnace cooling at a rate of 40°C/h up to 660°C/10h/ air cooling;

-Sample B - solution annealing to 950°C/1h/air cooling + double ageing at 771°C/8h/ furnace cooling at a rate of 40°C up to 660°C/10h/ air cooling;

-Sample C - solution annealing to 1080°C/1h/air cooling + ageing at 771°C/8h/ furnace cooling at a rate of 40°C up to 660°C/10h/ air cooling.

It can be noticed that the solution annealing treatment is performed at very high temperatures, for relatively long time periods, since the diffusion processes are conducted at very slow rates.

The alloy ageing treatment is carried out in two steps in order to obtain optimal quantity and dispersion of the hardening intermetallic compounds.

Conducted laboratory investigations

Optical microscopy analyses have allowed highlighting of carbides present in the alloy and grain size.

Electron microscopy analyses performed with a JEOL 100 CX/ASID 2D included energy dispersion analysis of micro-areas (EDX) to confirm the presence of niobium in the hardening precipitates, electron microscopy analysis on the two-steps replicas for morphology study of the hardening precipitates.

Sample preparation for transmission microscopy

A sample must be "transparently" to the electrons beam in order to be examined by transmission electron microscopy (TEM). There are several types of specimens that can be viewed using transmission electron microscopy: replica, foil or decorated samples. In the case of nickel-based superalloys replica-type samples are the most commonly used.

Replica-type samples preparation

Replica samples are used when the sample topography should be evaluated by high-resolution transmission; an electron diffraction analysis is requested for some precipitates or phases that occur in the sample matrix (extraction replica).

The method consists in depositing a thin layer of material on the surface of replica samples studied which then is separated from the sample, forming a negative replica of the sample relief, this being examined afterwards under TEM. Replicas are taken from the fracture areas or polished surfaces and are attacked by specific reagents to prominence the metallographic characteristics (phases, compounds etc).

There are several types of replicas, depending on the number of steps undertaken during processing:

- mono-step replicas in which the sample is detached from the very thin replica-film by chemical dissolution (atomic replica); this provides maximum resolution;
- mono-step replicas with thick film, when the film is mechanically detached from the original sample;
- two-steps replicas when the replica-film is coated with a carbon film and then with a hard metal (Pt, Rh, Au, Cr, etc.) film to shading by vacuum evaporation;
- extraction replica, obtained on heavily attacked samples with specific reagents, in order to erode the matrix without affecting the precipitates of interest that are extracted by capturing in the plastic material and examined through electron diffraction or other analysis methods provided by the electron microscopy (EELS, EDS etc).

All mentioned replicas must be shadowed to different angles for contrast improvement.

In this paper, the replicas method was used to identify the hardening precipitates present in the alloys; samples of Inconel 718 were mechanically polished and chemically attacked with the following mixture: ferric chloride (5 g), hydrochloric acid (10 mL) and methyl alcohol (100 mL).

A thin layer of nitrocellulose was deposited on the attacked and polished samples. After drying, the nitrocellulose was detached mechanically or by immersion in liquid nitrogen. The detached film was mounted on a glass slide with the mould part upward. A uniform carbon layer was deposited on the film thus prepared, using a vacuum deposition installation (HBA-2 type metalizing apparatus).

The carbon layer was perpendicularly deposited on the foil and was further covered with a germanium layer at a 45° angle (in order to obtain the contrast necessary for image analysis).

Results and discussions

The paper presents the results of electronic microscopy and diffractometer studies undertaken to identify and understand the evolution of characteristic phases present in the Inconel 718 superalloy.

Metallographic analysis allowed the determination of grain size; an Neophot optical microscope was used for this purpose.

Sample A presented adequate grains size (granulation 10). The same granulation tissue was obtained after sample B treatment [5].

Sample C showed a typical overheating aspect. Optical metallography studies have signalled large grain size (granulation 2 ... 3 on 90% of the analyzed surface, and granulation 10 on 10% of the analysed surface); a few types of carbides have been also noticed (squarely/ geometrically carbides TiC and most likely complex carbides - see reference [6]).

Figures 2 - 4 present optical microscopy images for samples B and C.

Figures 5 a and 5b present electron microscopy images of sample A collected from the Inconel 718 superalloy, analyzed with a JEOL 100CX microscope; the images captured strengthener precipitated (γ',γ'') in the austenitic matrix.

Identification and analysis of the strengthener precipitated (γ', γ'') (Ni_3Nb) was achieved by electronic microscopy analysis using electron micro-diffraction analysis and dark-field analysis.

Electron microscopy analysis was performed on a JEOL 100 CX transmission electron microscope (equipped with scanning analyser) at high resolutions; analysis on micro-areas was carried out by energy dispersive X-ray spectroscopy (EDX) to x 1200 magnification.

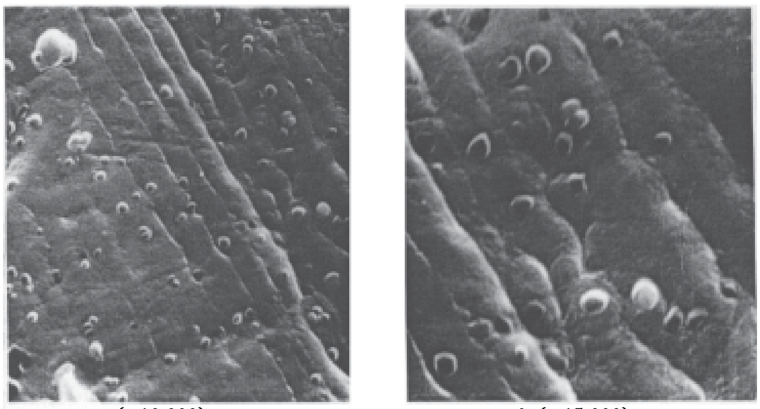
Figure 6 presents the micro-diffraction analysis result performed on sample A, where we have confirmation that the disc shape precipitates are Ni_3Nb type, so it precipitated as γ''.

Both images a) and b) in figure 7 are SEM electronic microscopy images; the c-image is a two step replica. These images were recorded on the sample B. In this case, Inconel 718 alloy has been kept for a longer time at the aging temperature.

γ''-precipitates tend to curdle in accordance with the phase transition (4), before forming the δ phase, which is a Ni_3Nb ordered phase, with an orthorhombic crystalline structure and lamellae form morphology.

The presence of Ni_3Nb orthorhombic crystalline precipitate is certificated by comparison with image 1325-1326 from reference [6]. This phase δ is not desirable, because its appearance leads to mechanical properties loss of the alloy. The resolution, which becomes visible in this δ phase, justifies the need for using specific electronic microscopy techniques when studying this material.

Fig. 2. Sample A; picture of optical microscopy (x2000)



a (x 10.000) b (x 15.000)

Fig. 5. Sample A; picture of electron microscopy SEI (scanning electron image)
a-Sliding plane with strengthener phasses γ' (γ").
b-Detail with strengthener phasses γ' (γ") in disk shaped at high resolution

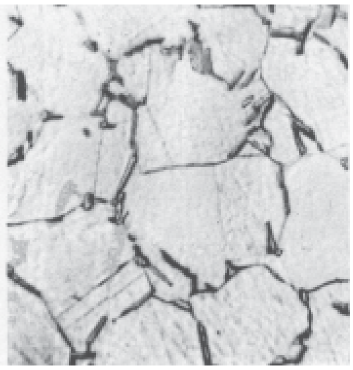


Fig. 3. Sample B; picture of optical microscopy (x2000)

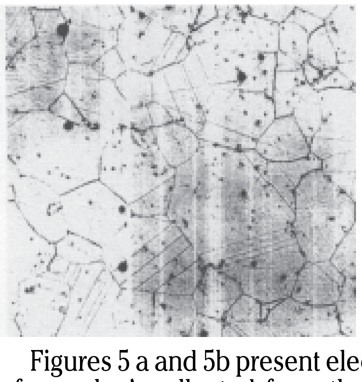


Fig. 4. Sample C; picture of optical microscopy (x200)

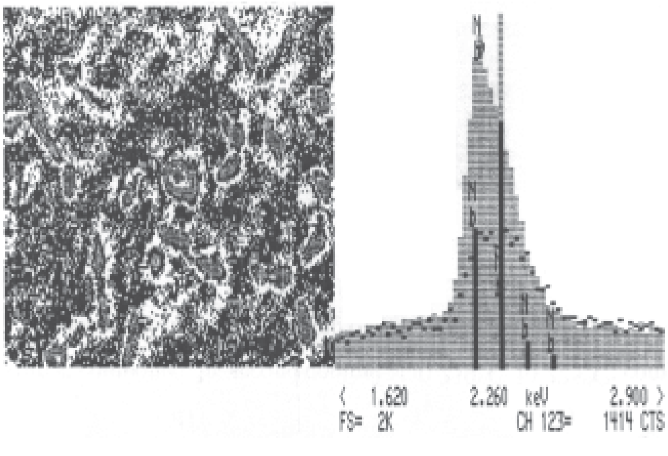


Fig 6. Qualitative results of morphology and elemental analysis (EDX) performed on a disc shape precipitate (Inconel 718 superalloy). Image scanning was carried out for x 1200 resolution

Fig. 7 a) Sample B SEI a (x 15.000)

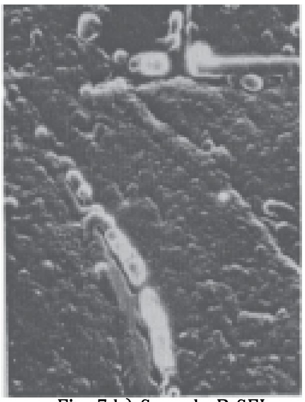


Fig. 7 b) Sample B SEI b (x 20.000)

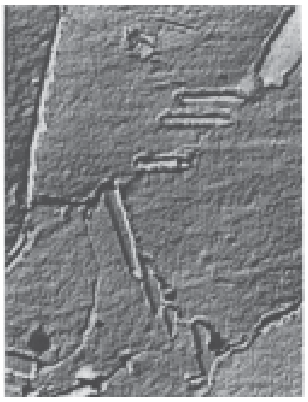


Fig. 7 c) Sample B SEI replica in two stages on c (x 3600)

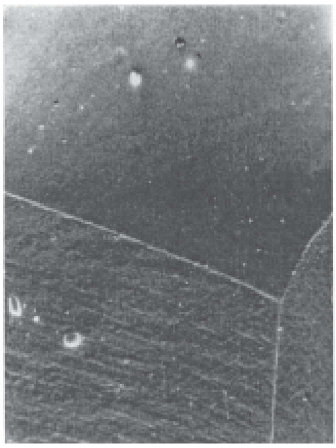


Fig.8 a) Sample C SEI (x2000)

Fig.8 b) Sample C SEI (x2000)

Images captured on the sample C, figure 8 a) and b), are typical overheating images; granulation is rough (noticed also in optical microscopy analysis). A large carbide, most likely titanium carbide, can be noticed in figure 8 b).

Conclusions

The transmission electron microscopy using the two-stage method and scanning analysis is an effective method to identify the phases and essential characteristics in superalloys with nickel base.

The strengthening agent in all heat-treated (solution and aging) 718 INCONEL superalloys is Ni_3Nb.

Appropriate heat treatment of this alloy grades consisted of solution annealing to 950°C/1h/air cooling + ageing at 771°C/8h/ furnace cooling at a rate of 40°C up to 660°C/ 10h/ air cooling.

The severe temperature limits required imply furnaces calibration and a strict delimitation of the areas capable to meet these requirements.

Heat treatment control of nickel-base superalloys can be carried out successfully by electronic microscopy analysis.

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