2011-36-0328

Study of the mechanical behavior of an Inconel 718 aged superalloy submitted to hot tensile tests

Felipe R. Caliari[1], Kátia C. G. Candioto[2, 3], Danieli A. P Reis[1], Antonio A. Couto[4, 5], Carlos de Moura Neto[1], Carlos A. Nunes[3]
[1]Instituto Tecnológico de Aeronáutica, São José dos Campos, SP, Brazil
[2]Associação Educacional Dom Bosco, Resende, RJ, Brazil
[3]Escola de Engenharia de Lorena–Universidade de São Paulo-DEMAR, Lorena, SP, Brazil
[4]Instituto de Pesquisas Energéticas e Nucleares, São Paulo, Brazil
[5]Universidade Presbiteriana Mackenzie, São Paulo, Brazil

ABSTRACT

This study aims to determine some mechanical properties of an Inconel 718 aged superalloy obtained through hot tensile tests. These properties as conventional 0.2% yield strength (σ_y), ultimate strength (σ_{UTS}), and specific elongation assessment (ε_u) are important parameters in the study of the mechanical material behavior. The superalloy was subjected to hot tensile tests at 650, to 700°C and a strain rate of 0.5 mm/min according to ASTM E-8. It is used a scanning electron microscope (SEM) to obtain images of the fracture surface of the specimens. The images of the fracture surface are analyzed in order to relate the temperature of the test with the type of mechanism of fracture.

INTRODUCTION

Inconel 718 is a precipitation-hardening superalloy developed by Internacional Nickel Co.[1] in the 50s. In 1989[4], this superalloy represented 45% of all wrought nickel-iron base superalloys produced in the world. It is used under high homologous temperatures ($T_h > 0.5$), showing high stress-rupture and good oxidation resistance, good creep and low cycle fatigue behavior. Superalloys are generally applied in heat treatment equipment, aeronautics gas turbines, nuclear power plants, medical components, chemical and petrochemical industries[2]. Inconel 718 has crystallographic lattices face-centered cubic (fcc), body-centered cubic (bcc), hexagonal close-packed (hcp) and body-centered tetragonal (bct), among others. This niquel-iron superalloys are made of austenitic fcc matrix γ (gamma phase), as well as secondary phases: gamma prime γ' face ordered $Ni_3(Al,Ti)$; gamma double prime γ" bct ordered Ni_3Nb; eta η hexagonal ordered Ni_3Ti; delta δ orthorhombic Ni_3Nb intermetallic compounds and other topologically closed-packed structures such as μ and Laves phases. δ, μ and Laves phases have low ductility, cause losses in mechanical and corrosion properties[2] and provide grain size control. In addition, these phases appear in alloys containing high level of bcc transition metals (Ta, Nb, Cr)[4] .

Secondary phases γ'and γ" play a main role in the strengthening mechanism of Inconel 718, mainly γ", a coherent disk-shaped precipitate. Changzeng et al. highlighted the structural stability dependence with secondary phases production on aging heat treatments, as well the competition of γ'/γ" and delta phase δ formation[3]. Superalloys have their microstructure characteristics improved by using heat treatment techniques. Solution treatment, usually the first step in heat treatment of precipitation hardening alloys, aims to: recrystallize, homogenize and dissolve phases in fcc matrix structure, dissolving carbides in grain boundaries and the grain-growth results in high creep-rupture resistance[4]. The purpose of aging treatments is the increasing of the strength through the precipitation of additional quantities of secondary phases, from the supersaturated matrix, developed by solution treating. For Inconel 718 more than one phase can precipitate, so double aging is applied in order to enhance the formation of both γ'and γ" phases.

MATERIAL AND EXPERIMENTAL METHODS

Material was provided in bars by Multialloy. The specimens were machined by Fautec Ferramentaria Automação e Usinagem Ltda. The superalloy was obtained by VIM/VAR process. The heat treatment was conducted at EEL/DEMAR, using a Lindberg/Blue M - Tube Furnace 100V/50A/5kW and followed the sequence described on table 1.

Table 1: Heat treatment condition applied to Inconel 718.

Heat treatment step	Parameters
Solid Solution	1095ºC; 1h/AC
Double Aging	955ºC; 1h/AC
	720ºC; 6.5h/FC
	720ºC; 1.5h/FC
	620ºC; 8h/AC

The composition of the superalloy used in this study is summarized on table 2.

Table 2: Inconel 718 composition.

Analyse %wt										
C	**S**	**Cr**	**Ni**	**Mn**	**Si**	**Mo**	**Ti**	**Nb**	**Cu**	**Bi**
0.03	<0.002	18.39	52.83	0.03	0.03	3.01	0.95	5.05	0.03	<0.00003
Fe	**P**	**Al**	**Mg**	**Pb**	**Ca**	**Co**	**B**	**Ta**	**Se**	
18.87	0.004	0.48	<0.001	0.0002	0.001	0.23	0.001	0.01	<0.00003	

HOT TENSILE TEST

The uniaxial hot tensile test was carried out using Instron 3382 100 kN Metal Package equiped with Instron's Series IX^{TM}/s software, under temperatures of 650ºC and 700ºC, at constant elongation-rate of 0.5 mm/min, according to ASTM E-8[10]. The specimen condition is set up as showed in figure 1.

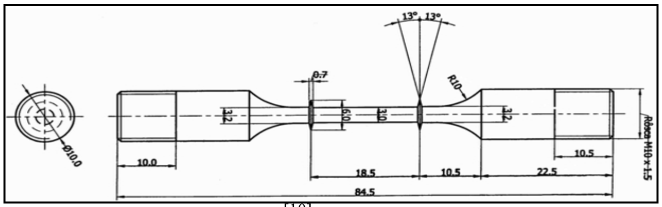


Figure 1 – Specimen dimensions[10].

METALOGRAPHIC PREPARATION

The specimens were prepared by conventional metallographic procedures (transversal sectioning followed by mounting in bakelite). Grinding procedure was conducted using 180, 600 and 1200 grits. Polishing was carried out with cloth covering 6 µm impregnated with 6 µm diamond paste. After that the specimen was polished using a 1 µm cloth covering with 1 µm diamond paste impregnated. Both grinding and polishing procedures were performed on METPREP 3 PH-3 (Allied High Tech products) equipment. Between every procedure the specimens were submitted to ultrasound cleaning, 5 minutes each time. Electrochemical etching was performed by oxalic acid 10% solution, over 20 seconds and using 5V voltage.

For the fractography analysis, the fractured specimen was cleaned using ultrasound equipment, immersed in acetone solution during 5 minutes.

RESULTS AND DISCUSSION

RESULTS

Hot tensile results

In this study mechanical properties of Inconel 718 before and after double aging treatment are compared. Engineering stress – strain curves of as received Inconel 718 are shown in figures 2 and 3; double aged Inconel 718 engineering stress – strain curves are presented in figures 4 and 5. Table 6 shows summary of microhardness (Vickers) values for Inconel 718 before and after heat treatment.

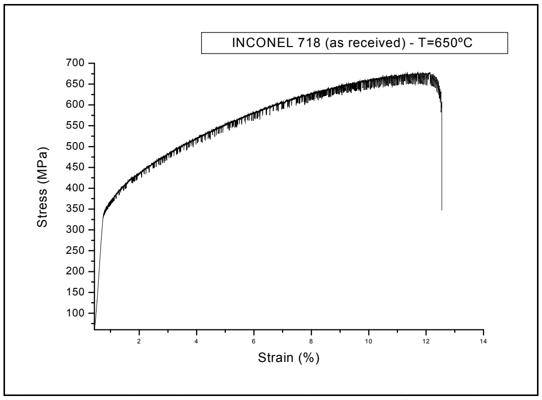


Figure 2 – Engineering stress – strain curves of Inconel 718 (without heat treatment) at 650ºC

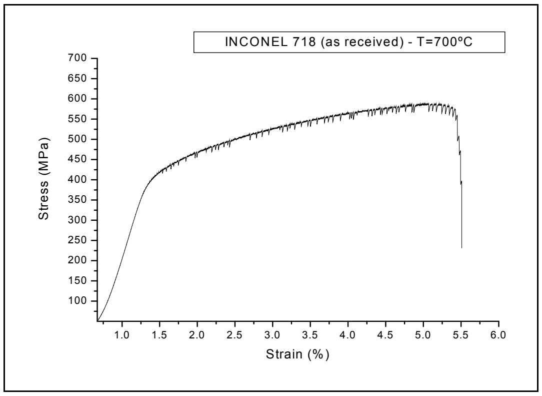


Figure 3 – Engineering stress – strain curves of Inconel 718 (without heat treatment) at 700ºC

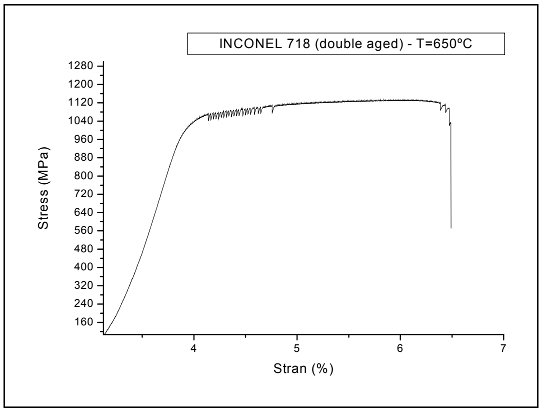


Figure 4 – Engineering stress – strain curves of Inconel 718 (double aged) at 650ºC

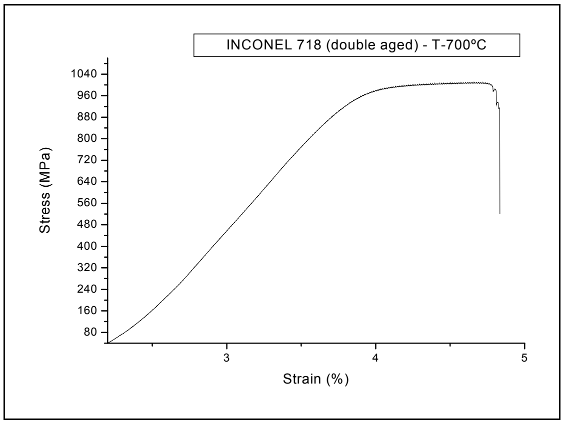


Figure 5 – Engineering stress – strain curves of Inconel 718 (double aged) at 700ºC.

A summary of the superalloy mechanical properties, in as received and double aged states, are shown in tables 3 and 4, respectively.

Table 3: Inconel 718 properties: as received condition.

Inconel 718 (As received)		
Temperature [ºC]	**650**	**700**
0.2% offset yield strength (σ_y) [MPa]	355.35	421.48
Ultimate strength (σ_{UTS}) [MPa]	676.32	588.56
Specific elongation at failure (ε_u) [%]	12.55	5.51

Table 4: Inconel 718 properties: after double aging heat treatment.

Inconel 718 (double aged)		
Temperature [ºC]	650	700
0.2% offset yield strength (σ_y) [MPa]	1062.61	974.31
Ultimate strength (σ_{UTS}) [MPa]	1130.24	1006.50
Specific elongation at failure [%]	6.49	4.83

Table 5 Inconel 718 reduction area after hot tensile tests

Reduction in area [%]		
As received	T=650ºC	28.32
	T=700ºC	12.27
Double aged	T=650ºC	6.77
	T=700ºC	2.45

Table 6: Comparison between microhardness values of Inconel 718 before and after heat treatment.

Inconel 718 condition	Average values – Microhardness [HV]
As received	176.40
Double aged	459.26

SEM image results

Scanning electron microscopy technique is used to evaluate qualitatively the superalloy microstructure, along with the Energy-dispersive spectrometry. In order to reveal the metallographic images (figures 6 to 11), backscattered electrons signal was applied. EDS technique helps to identify chemical composition of secondary phases. An EDS of the precipitate showed in figure 7 can be found on figure 12, and the EDS of figure 8 is detailed in figure 13. A LEO 1450 VP scanning electron microscope from EEL/DEMAR, equipped with Oxford Instruments energy-dispersive X-ray spectrometry system was used in this work. Figure 6 shows a general view (300 X) of non-heat treated Inconel 718 microstructure and figures 7 and 8 are detailed views of the secondary phases.

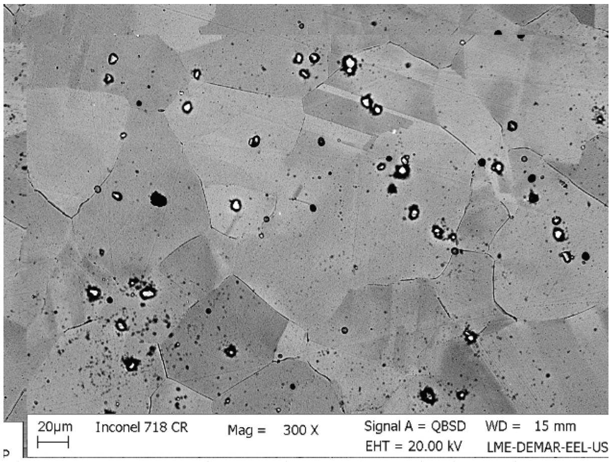


Figure 6 – SEM of Inconel 718 (general view of an as received microstructure).

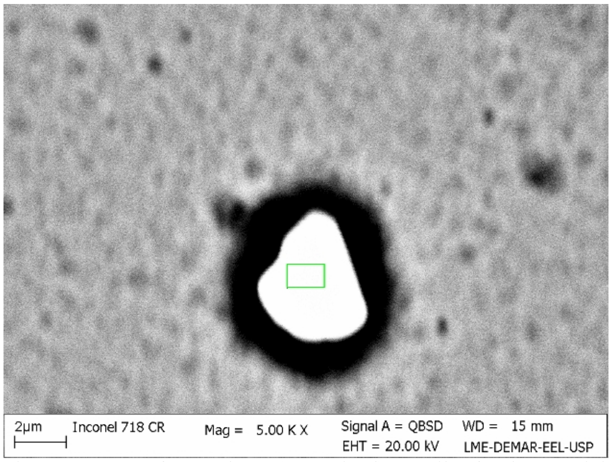


Figure 7 – SEM of Inconel 718 (as received): Details of a clear precipitate.

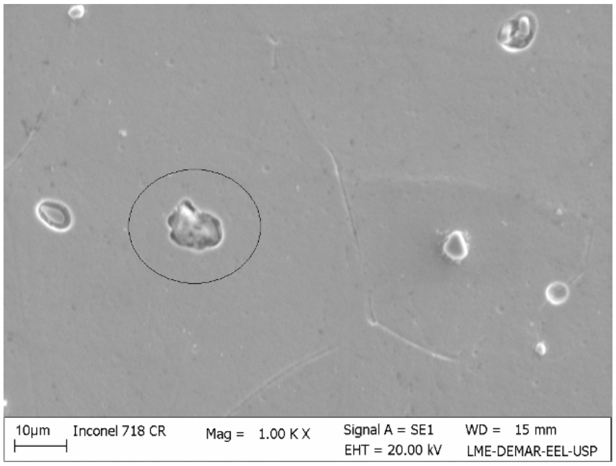


Figure 8 – SEM of Inconel 718 (as received): Details of a dark precipitate.

The following figures (9 to 11) present the double aged Inconel 718 microstructure. Figure 9 shows a general image (300 X) of the superalloy microstructure. Figures 10 and 11 detail the precipitates found during SEM analyzes. From figure 9 it can observe the grain growth resulted from aging treatment and a better dispersion of secondary phases. EDS of precipitates found in pictures 10 and 11 can be observed in figures 14 and 15, respectively.

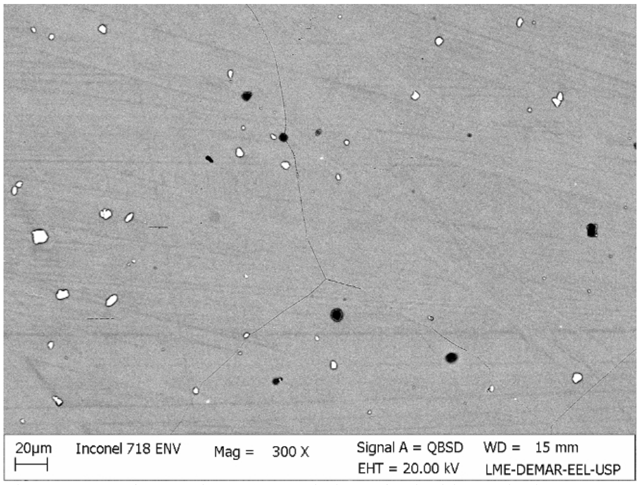


Fig 9 – SEM of Inconel 718 (general view of a double aged microstructure).

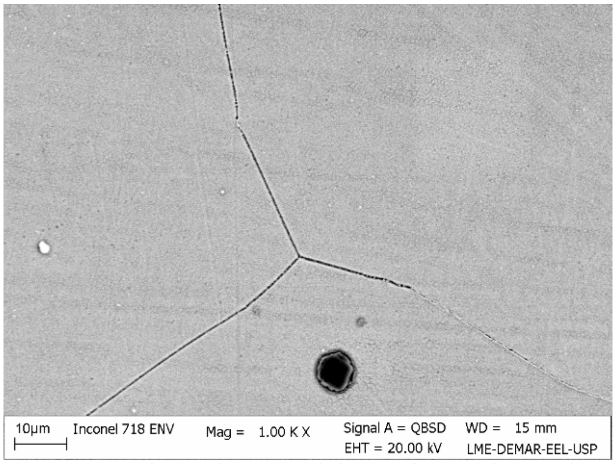


Fig 10 – SEM of Inconel 718 (detailed view of a precipitate on the matrix of double aged Inconel 718).

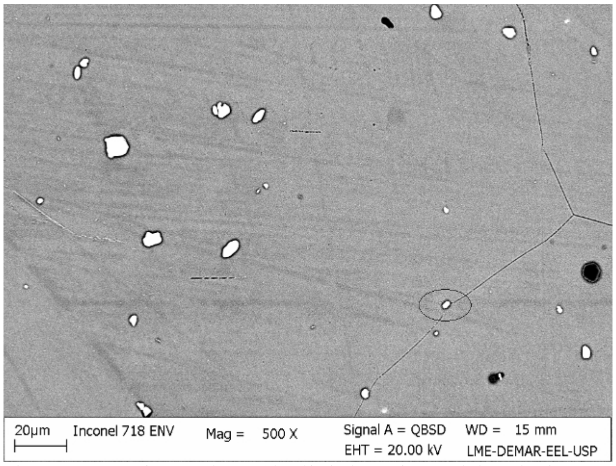
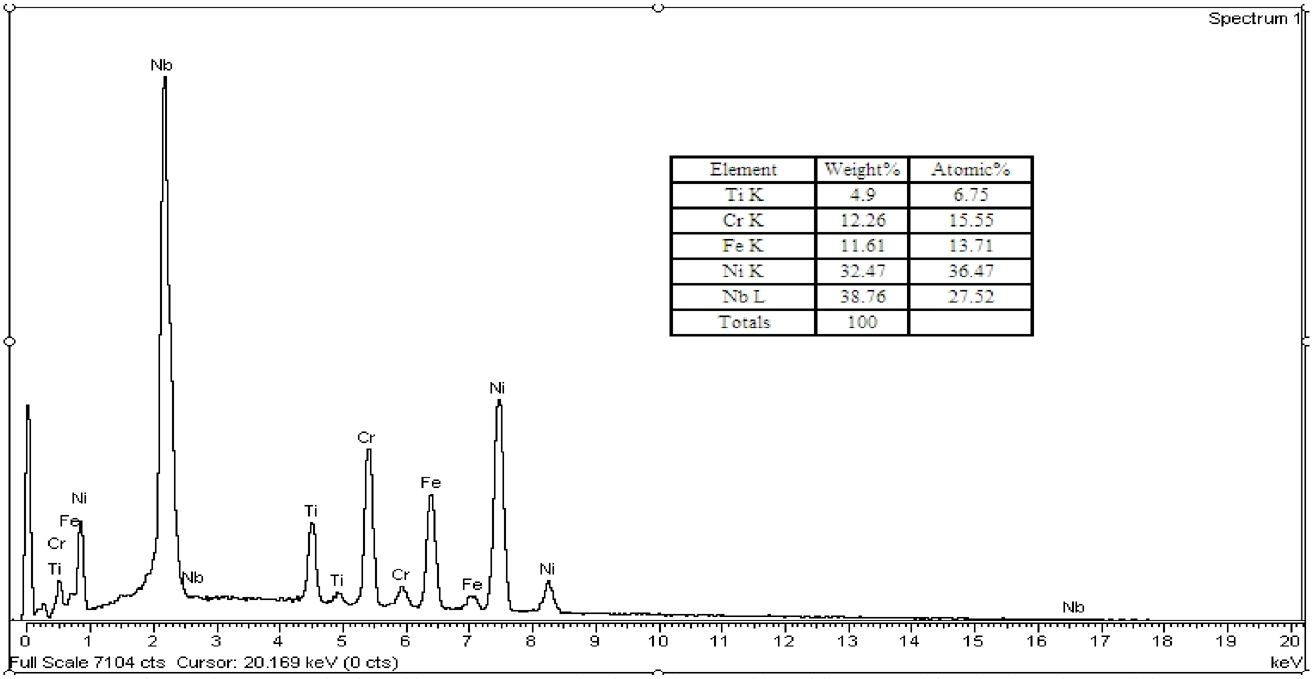
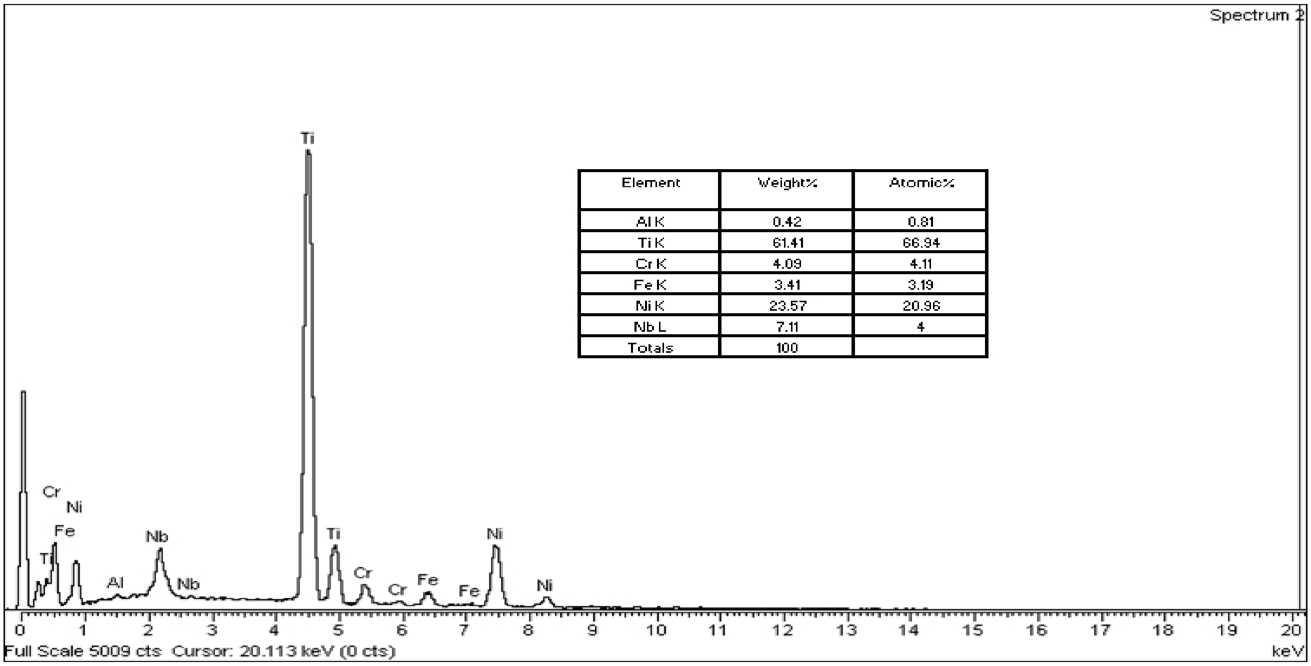


Fig 11 – SEM of Inconel 718 (detailed view of a precipitate in the grain boundary of double aged Inconel 718).



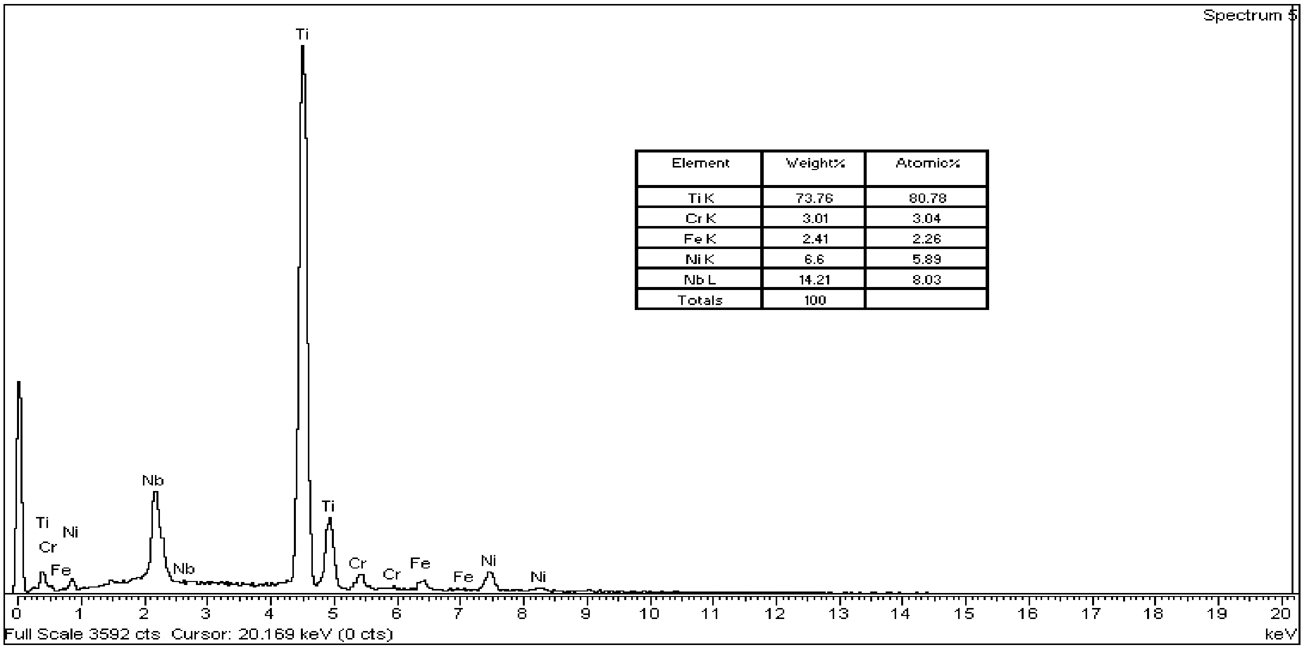
Element	Weight%	Atomic%
Ti K	4.9	6.75
Cr K	12.26	15.55
Fe K	11.61	13.71
Ni K	32.47	36.47
Nb L	38.76	27.52
Totals	100	

Fig 12 – SEM of Inconel 718 (as received) – This EDS represents the chemical composition of figure 7, and found to be a clear precipitate.



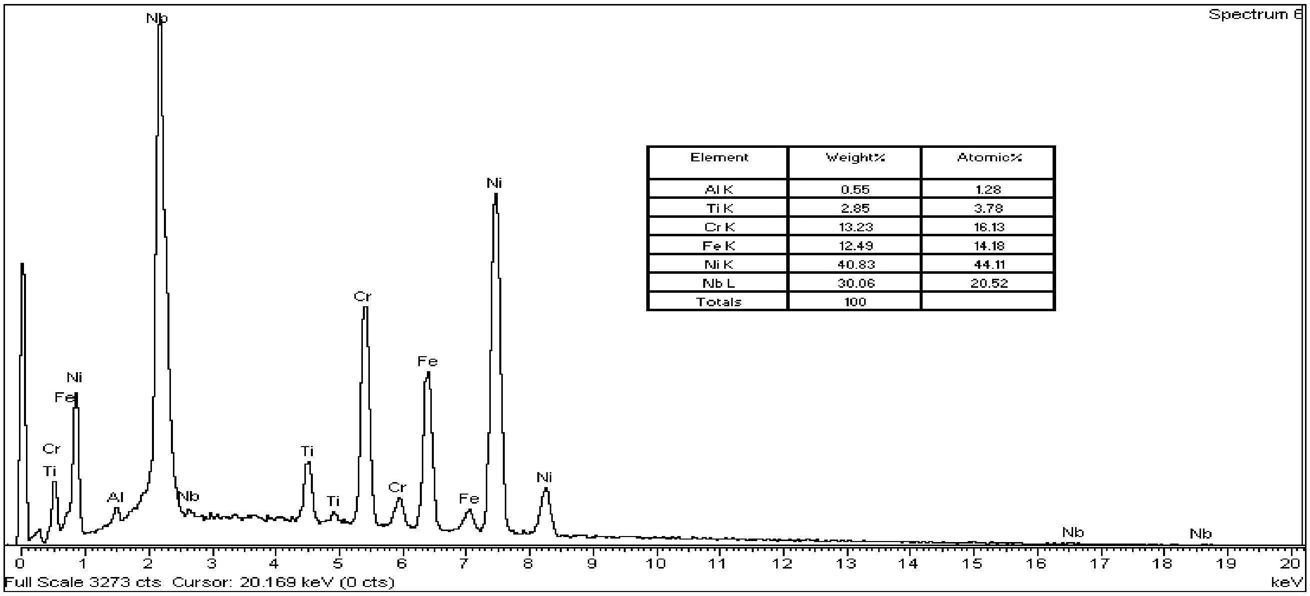
Element	Weight%	Atomic%
Al K	0.42	0.81
Ti K	61.41	66.94
Cr K	4.09	4.11
Fe K	3.41	3.19
Ni K	23.57	20.96
Nb L	7.11	4
Totals	100	

Fig 13 – SEM of Inconel 718 (as received) – This EDS represents the chemical composition of figure 8, and found to be a dark precipitate.



Element	Weight%	Atomic%
Ti K	73.76	80.78
Cr K	3.01	3.04
Fe K	2.41	2.26
Ni K	6.6	5.89
Nb L	14.21	8.03
Totals	100	

Fig 14 – SEM of Inconel 718 (double aged) – This EDS represents the chemical composition of figure 10 The precipitate is located in the matrix.



Element	Weight%	Atomic%
Al K	0.55	1.28
Ti K	2.85	3.78
Cr K	13.23	16.13
Fe K	12.49	14.18
Ni K	40.83	44.11
Nb L	30.06	20.52
Totals	100	

Fig 15 – SEM of Inconel 718 (double aged) – This EDS represents the chemical composition of figure 11. The precipitate is located on grain boundary.

Fractography image results

In order to reveal the fractography images (figures 16 to 27), SEM technique with secondary electrons signal was applied. The results can be found on the following pictures (Figures 16 to 27).

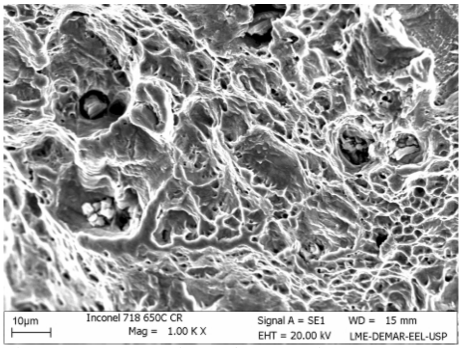


Fig 16–Fractography of Inconel 718 (as received), T=650ºC (center of the specimen).

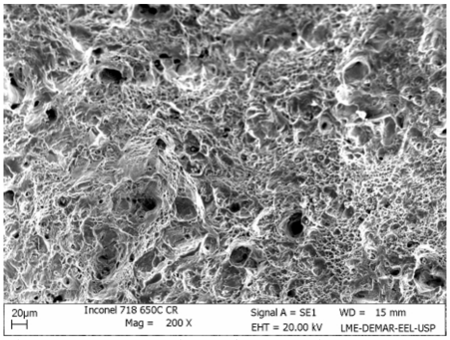


Fig 17–Fractography of Inconel 718 (as received), T=650ºC (edge of the specimen).

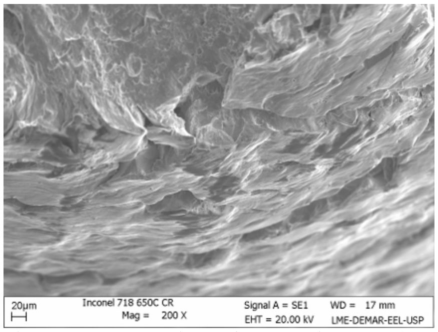


Fig 18–Fractography of Inconel 718 (as received), T=650ºC (sideview of the specimen).

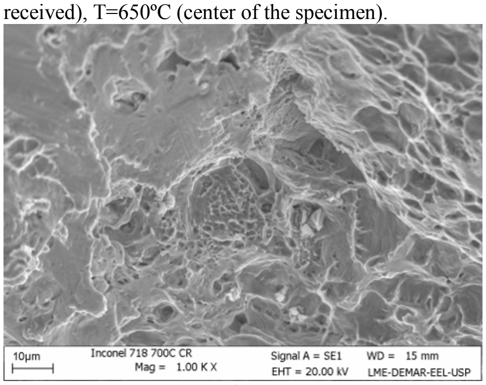


Fig 19–Fractography of Inconel 718 (as received), T=700ºC (center of the specimen).

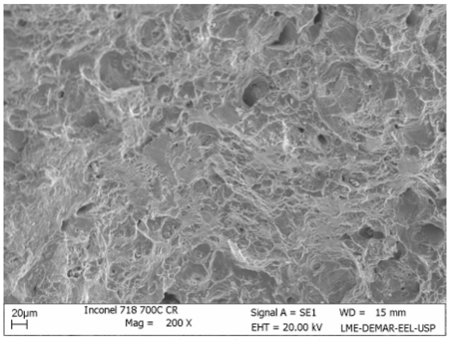


Fig 20–Fractography of Inconel 718 (as received), T=700ºC (edge of the specimen).

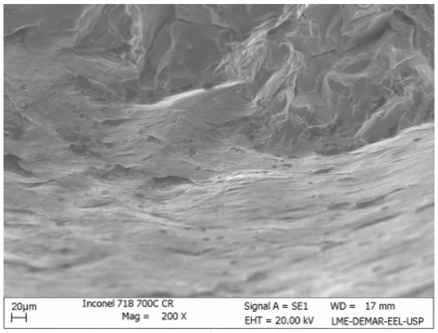


Fig 21 –Fractography of Inconel 718 (as received), T=700ºC (sideview of the specimen).

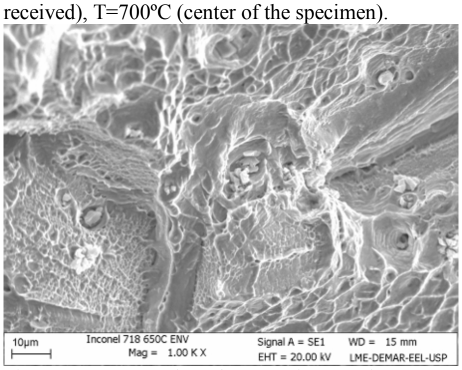


Fig 22 –Fractography of an aged Inconel 718, T=650ºC (center of the specimen).

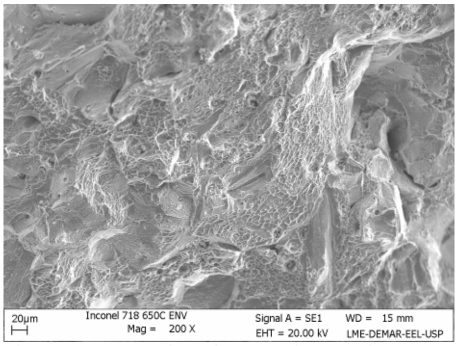


Fig 23–Fractography of an aged Inconel 718, T=650ºC (edge of the specimen).

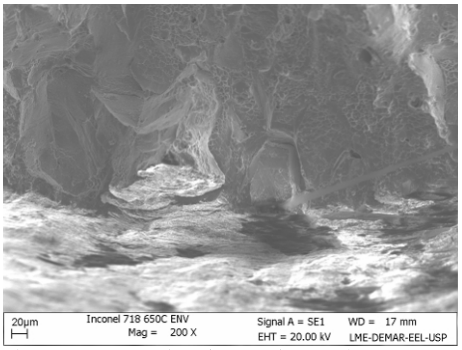


Fig 24 –Fractography of an aged Inconel 718, T=650ºC (sideview of the specimen).

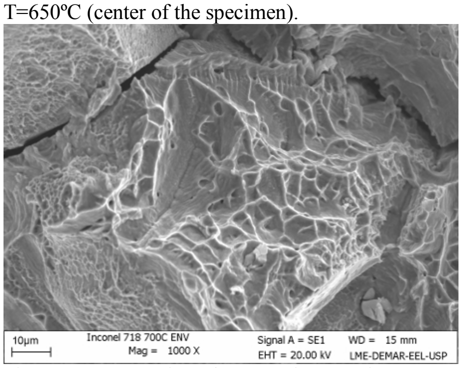


Fig 25–Fractography of an aged Inconel 718, T=700ºC (center of the specimen)

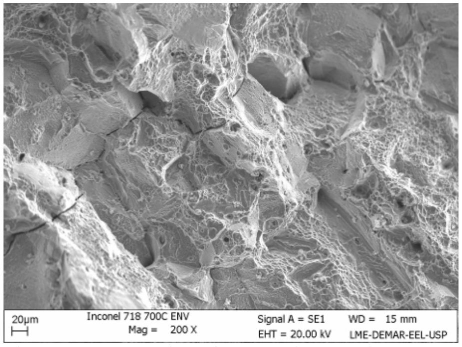


Fig 26–Fractography of an aged Inconel 718, T=700ºC (edge of the specimen)

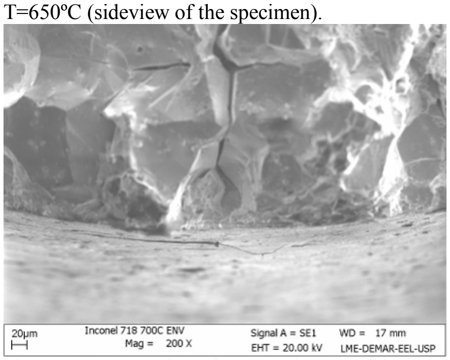


Fig 27–Fractography of an aged Inconel 718, T=700ºC (sideview of the specimen)

DISCUSSION

Inconel 718 as received:

As can be seen on figures 2 and 3, from 650ºC to 700ºC the percentual elongation has decreased and the yield strength increased about 16%; but ultimate strength has decreased 15%. Increasing yield strength may be due to the presence of γ’ phase which can be formed via heating process in hot tensile test. According to Thomas et al.[8] secondary phases γ’and γ” can be found in less extension below 700ºC. This observation can explain the drop of the ultimate strength.

Engineering stress – strain curves of Inconel 718, in the as received condition, shows the Dynamic Strain Aging (DSA)[6] phenomenon. In order to this effect happen, it is necessary to lower the content of carbon in solution, adding typically interstitial elements which will form stable carbides. Addition of aluminum, vanadium, titanium, niobium and boron end up producing the latter mentioned stable carbide forming[7]. The serration behavior in figures 2 and 3 is explained by the Portevin-Le Chatelier effect, which is related to dynamic equilibrium of yield strength and dislocations barrier energy. Results of SEM/EDS analyze (figures 6 to 10) show details of secondary phases distribution along γ matrix. One can notice, in figure 6, the non-homogeneous distribution of precipitates. According to EDS results (fig. 9 and 10), figures 7 and 8 correspond to precipitates based on Ni/Nb (γ”) and Ni/Ti (γ’).

As received Inconel 718 fractography

The fractography of the as received Inconel 718 is shown on figures 16 to 21. According to the figures 16 to 18, at 650ºC the main fracture mechanism is an intergranular dimple rupture, which is produced by microvoids nucleation observed on localized strain discontinuity, and it is typically characterized by cuplike depression[11]. This fracture mode is associated mainly with secondary phases, and dislocation pile-ups. Comparing to the prior situation, the fracture mechanism of Inconel 718 (as received) at 700ºC shows brittle characteristics, and it has also a cleavage fracture behavior, due to presence of flat planes. In both situations precipitate particles can be seen inside dimple cavities, which are nucleation sites for the holes.

Inconel 718 double aged:

The explanation of stress – strain results relies on precipitation strengthening mechanism, induced by solubilization and followed by aging treatment. As can be seen from tables 3 and 4 and corresponding graphics 4 and 5 , the yield strength of Inconel 718 has increased from 2 to 3 times comparing to the non aged alloy, and the specific elongation decreased about 50% considering the temperature of 650ºC. The value of 1,062.61 MPa (see table 4, temperature of 650ºC and same heat treatment from reference) is coherent with literature reference value of 1020 MPa[9] Comparing the aged superalloy test results on hot tensile 650 and 700ºC tests, it appears a small decrease in toughness. This behavior can be associated with the presence of a δ phase[5], which probably have raised up in the first aging treatment stage (955ºC ,1h), although the solution treating was performed above the *solvus* temperature of delta phase. In figure 9 it, compared to figure 6, it can be seen a grain-growth and a better dispersion of secondary phases. A detailed EDS investigation on precipitates found on double aged superalloy can be observed on figures 14 and 15. Results of figure 14 (see figure 10) show a precipitate based on Ti/Nb/Ni (not specified precipitate). The EDS of figure 15 (see image on figure 11) shows a precipitate on grain boundary, with main composition of Ni/Nb (possibly delta phase δ). Further, microhardness (Vickers) results reinforce the effectiveness of heat treatment in the re-distribute secondary phases and in the strengthening mechanism.

Double aged Inconel 718 fractography

Double aged Inconel 718 shows cleavage brittle-fracture behavior on fractography according to SEM images, along with small contribution of dimple fracture mechanism. Figures 22 to 26 detail image of fractured material. On figures 25 and 26 it is seen a clear actuation of transgranular fracture path. In that case Inconel 718 has been tested at 700ºC.

SUMMARY/CONCLUSIONS

Deformation under hot tensile tests of Inconel 718 had distinguished behavior comparing the as received and double aged state. Heat treatment of double aging is capable to reorganize the microstructure of the precipitates and produce a three times strengthen yield point. Results of fractography images demonstrate a transition from intergranular dimple rupture to a transgranular cleavage fracture, after heat treatment.

Further studies of creep resistance are necessary in order to establish the superalloy double aged broader behavior under high homologue temperature and stresses.

REFERENCES

1. Silva, André Luiz V. da Costa; Mei, Paulo Roberto. Aços e ligas especiais. São Paulo: Blücher, 2010. Cap. 7, p. 450-458.
2. M. Donachie, Jr., S. Donachie. Superalloys: A technical guide – 2nd ed. ASM International.
3. Chanzeng Wang, Rongbin Li. Effect of double aging treatment on structure in Inconel 718 alloy. Journal of Material Science 39 (2004), p. 2593-2595.
4. American Society for metals. ASM Handbook, v. 4, 1994, p. 793-814, 907-912.
5. Said Azadian, Liu-Yingwei, Richard Warren. Delta phase precipitation in Inconel 718. Materials Characterization 53 (2004), p. 7-16.
6. Kartik Prasad, Rajdeep Sarkar, P. Ghosal, Vikas Kumar. Tensile deformation behaviour of forged disk of Inconel 718 superalloy at 650ºC. Materials and Design (2010).
7. Dieter, George E.; Mechanical Metallurgy – SI edition. McGraw-Hill. 1998.
8. A. Thomas, M. El-Wahabi, J.M. Cabrera, J.M. Prado. High temperature deformation of Inconel 718. Journal of Materials Processing Technology 177 (2006), p. 469-472.
9. American Society for Metals. ASM Handbook, v. 1, 1993,
10. ASTM E-8, Standard Test Methods for Tension Testing of Metallic Materials. American Association State Highway and Transportation Officials Standard - Annual Book of ASTM Standards (2004).
11. American Society for Metals. ASM Handbook, v. 12, 1994, p. 793-814, 907-912

CONTACT INFORMATION

Felipe Rocha Caliari
MSc student – ITA/DCTA, São José dos Campos/SP
e-mail: felipercaliari@yahoo.com.br
tel: +55 12 3947-5876

ACKNOWLEDGMENTS

ITA
DCTA
ipen
EEL-USP
Escola de Engenharia de Lorena

DEFINITIONS/ABBREVIATIONS

T_h – Homologous temperature
SEM – Scanning electron microscopy
EDS – Energy dispersive spectrometry