Investigation of low stress rupture properties in Inconel-718 super alloy

T. Zaman, M. Farooque, S. A. Rizvi, I. Salam and M. Waseem
Institute of Industrial Control Systems, P. O. Box 1398, Rawalpindi, Pakistan

E-mail: ishmaelnn@gmail.com

Abstract. Inconel-718 is a Ni-Cr-Fe based super alloy. It is widely utilized in aircraft gas turbines, nuclear power systems, space vehicles and medical applications. Aim of the present study is to evaluate the effect of Ti and Nb content on high temperature stress rupture properties of Inconel718. OM, SEM and TEM were utilized for characterization of microstructure. Inconel718 is unique in that it forms large number of phases due to its composition and variety of heat treatments. γ''+ γ' precipitates and the effect of annealing on these precipitates have been studied using TEM. The main hardening phase was identified as metastable Ni3Nb (γ''). Other phases identified after annealing were secondary carbides (NbC) and stable acicular δ phase. Effect of γ'', δ, primary carbides and NbC on creep behavior was observed using OM and SEM. Higher Ti content(1.25 wt. %) resulted in poor creep properties due to large concentrations of primary carbides (TiC) at grain boundaries.

1. Introduction

Inconel718 is a nickel-based superalloy having good corrosion resistance and excellent mechanical properties at high temperatures. These alloys are particularly preferred in applications requiring good creep properties. These superalloys are extensively used as disc material in gas turbines and jet engines where service temperatures can be as high as 650°C [1-4].

Carbides play a very critical role in superalloys. Hence, studying the morphology, behavior and nature of these carbides is very important. Molybdenum, chromium, niobium, vanadium and titanium are carbide forming elements. MC, $M_{23}C_6$, M_6C and M_7C_3 type carbides are usually observed in superalloys [5]. Composition of alloy,heat treatment and deformation parameters dictate the morphology of these carbides. Inconel718 has an austenitic (γ) matrix. γ' and γ'' are the main strengthening phases of inconel718 superalloy. γ' is Ni_3Al while γ'' is Ni_3Nb. In full heat treated condition i.e. after solution treatment and double aging, the %volume fraction of γ'' and γ' is 20% and 5% respectively [6-7].

Xishan Xie et al studied the effect of titanium, niobium and aluminum on precipitation and strengthening behavior of 718 type superalloys [8]. They studied the characteristics of γ' and γ'' phase using scanning electron microscope and transmission electron microscope. They also determined the weight fraction of γ' + γ'' phases by electrolytic isolation followed by micro-chemical analysis. Moukrane Dehmas et al. utilized transmission electron microscope to study the high temperature precipitation of δ phase in inconel718 alloy [1]. They investigated a case where δ phase precipitated directly from the austenitic (γ) matrix. Presence of rotation-ordered domains in δ-plates was also confirmed.

Aim of this study was to investigate the influence of titanium content on high temperature stress rupture properties of Inconel718 superalloy. Secondly the effect of heat treatment on different precipitated phases (γ′, γ″, δ, metallic carbides) was also studied using scanning transmission electron microscopy.

2. Experimental

Inco718 rod (diameter: 12mm) was utilized for this investigation. Chemical composition of the rod is given in Table 1. Chemical composition was obtained using Optical Emission Spectrometer (OES). Mechanical testing of the rod included: Hardness testing, tensile testing and creep testing.
Tensile testing was carried out using INSTRON 1195. For creep testing ATF-2330 creep tester was employed. Tests were conducted at 649°C at a stress level of 690 MPa.

Table 1. Chemical composition weight %

Elements	Ni	Cr	Fe	Nb	Mo	Ti	Al	(Al+Ti+Nb)at.%
ASTM B637-03	50-55	17-21	17-18	4.7-5.5	2.8-3.3	0.6-1.15	0.2-0.8	5.5
Composition wt.%	53	18.87	17.19	5.5	3.06	1.25	0.52	5.98

Samples were prepared before and after creep testing from cross-sectional and longitudinal regions. Samples were ground using180 to 1200 grit silicon carbide paper.1µm diamond paste was utilized for final polishing. Electrolytic etching of Inco718 samples was done with 10% oxalic acid. Average grain size was recorded using mean linear intercept method. Optical microscope OLYMPUS BX51 was used for studying polished and etched samples.

Fractured surfaces after high- temperature creep testing were observed using Scanning Electron Microscope JEOL JSM5910LV equipped with Energy Dispersive Spectrometer (EDS).
Scanning Transmission Electron Microscope JEOL-JEM-2000FXII was employed for study of precipitated phases in Inconel718. 3mm discs were manually punched out and ground using SiC paper, till final thickness of 0.1mm was achieved. These disc samples were then electrolytically prepared in Twin Jet Electropolisher using perchloric acid electrolyte (at 23V). Temperature was kept at approximately -15°C.

3. Results and discussion

3.1 Mechanical testing

Table 2 presents results of hardness and tensile testing. Yield strength of 1292MPa and ultimate tensile strength of 1512MPa was observed. %Elongation and % reduction in Area were recorded as 17.8% and 34.5% respectively. Hence, tensile properties of Inconel718 rod were well within the range of ASTM B637-03 standard.

Table 2. Mechanical properties: room temperature

Properties	Y.S (MPa)	UTS (MPa)	% Elong.	%Red. of Area	Hardness(HB)
ASTM B637-03	> 1034	> 1275	> 12	> 15	> 331 HB
Inconel718	1292	1512	17.8	34.5	434

Table 3 depicts results of creep testing done at 649°C (690MPa). Creep specimens sustained for total time of 80.3 hours before finally breaking. %Elongation observed in sample after 23 hours of testing was 1.07%. This %elongation is below the minimum required range of 4% (according to ASTM B637-03). A total %elongation of 4.24% was recorded in the sample after 80.3 hours. In order to investigate the root cause of low stress rupture value, chemical composition was observed which showed that the titanium content in rod was 1.25 weight %. This content is higher than the maximum permissible limit of 0.6-1.15(weight %). This abnormality in chemical composition formed the basis of investigation.

Table 3. Mechanical properties: high temperature

Properties	**Temp.**	**Stress**	**Time Sustained hrs.**	**%E.(23 hrs.)**	**Total %E**
ASTM B637-03	649°C	690 MPa	> 23	> 4%	--
Inconel718	649°C	690 MPa	80.3	1.07	4.24

(Al + Ti + Nb) atomic % is reported to be 5.5 atomic % for Inconel718 alloys [9]. However, in this study a higher (Al + Ti + Nb) atomic % of 5.98 atomic% was observed. Aluminum, titanium and niobium play key role in determining mechanical properties of Inconel718 alloy. These elements combine with carbon in order to give various metallic carbides. These metallic carbides are subdivided into: primary carbides (e.g. TiC) and secondary carbides (e.g. NbC).

3.2 Metallic carbides
Polished samples were studied under optical microscope in order to reveal metallic carbides. Metallography samples were prepared both before and after creep testing. Figure 1 shows TiC in polished Inconel718 alloy after creep testing. Average size of these primary carbides was recorded to be 5-6µm. these samples were then observed under Scanning Electron Microscope. SEM revealed a large quantity of titanium and niobium carbides located at the grain boundaries. Figure 2 shows primary carbides before creep testing (left) and after creep testing (right). It was observed that size of metallic carbides increased after creep testing. In terms of morphology, blocky/cube shaped, spherical and irregular carbides were mainly observed. The presence of primary carbides at grain boundaries effect high temperature stress rupture properties.

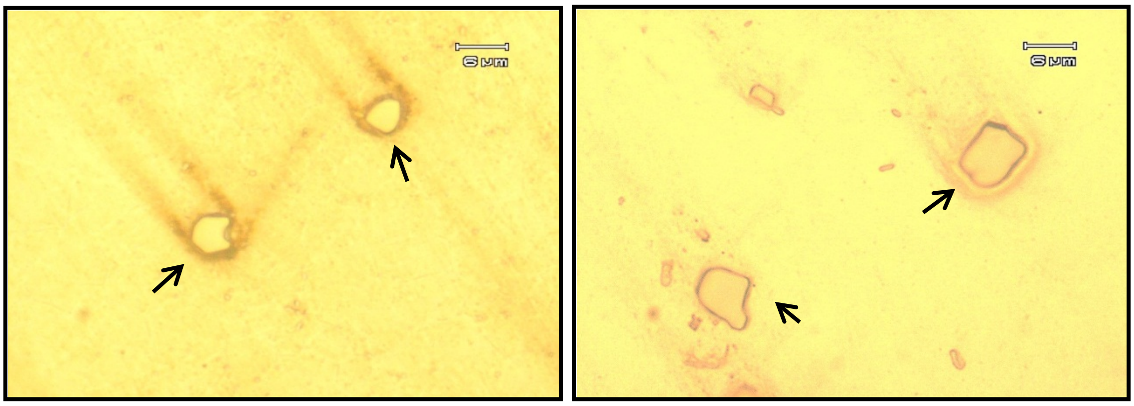

Figure 1. Optical micrographs of primary carbides (TiC); after creep testing

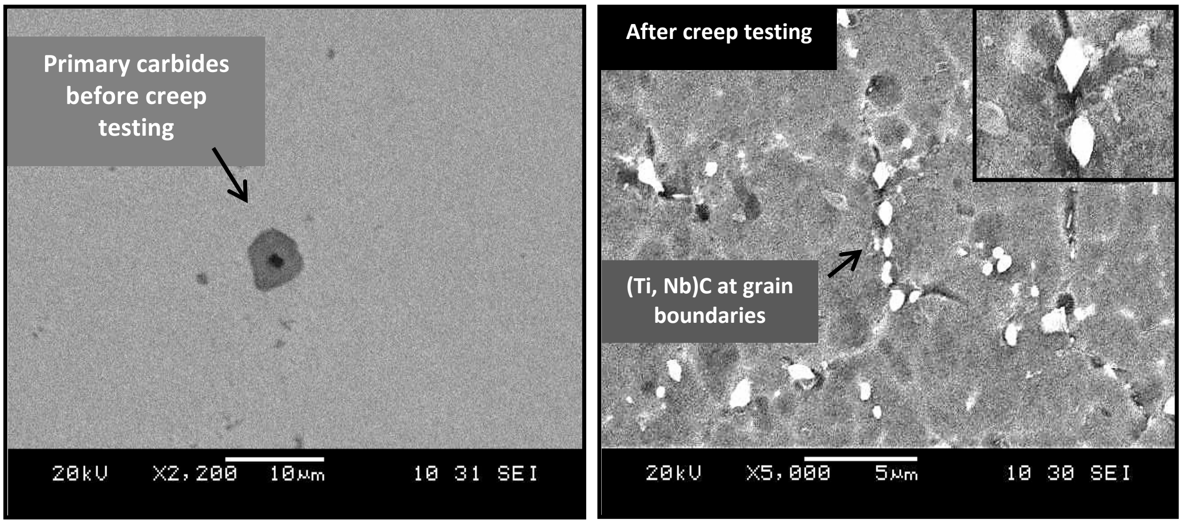

Figure 2. SEM micrographs depicting metallic carbides located at grain boundaries; before creep test (left) and after creep test (right)

3.3 Fractography

Figure 3 depicts Scanning Electron Microscope images of specimens fractured during high temperature creep testing. Microvoid coalescence phenomenon was dominant indicating ductile mode of failure. A large quantity of primary carbides was observed in the fractured surface. Presence of these carbides led to nucleation of primary micro-voids. Presence of large primary carbides of 5-6µm at grain boundaries deteriorated creep strength of inconel718 rod. This large concentration of PCs (TiC etc.) was actually due to higher titanium content (1.25 weight %) in the rod. Thus composition plays critical role in determining mechanical properties of a material. By regulating the titanium, niobium and aluminum content, the final mechanical properties of Inconel718 alloy can be controlled to a great extent.

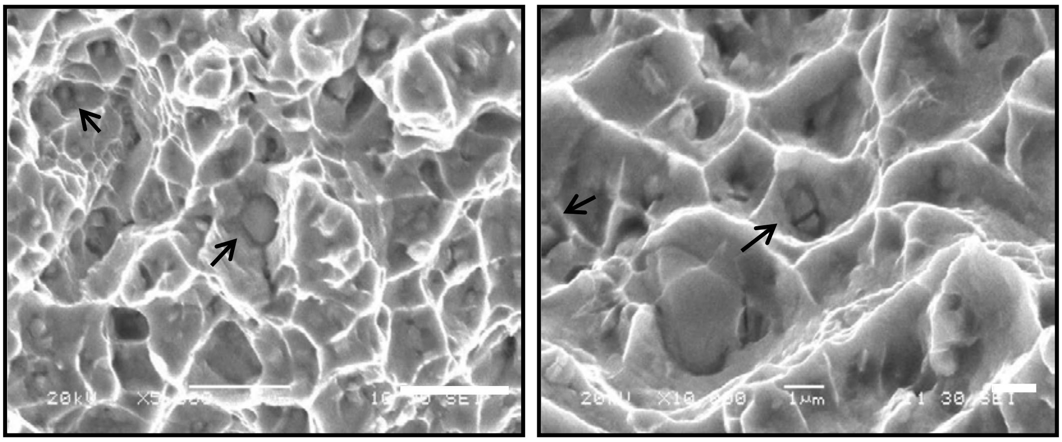

Figure 3. SEM fractographs depicting primary micro-voids nucleated at sites of (Ti, Nb)C

3.4 Precipitated phases

Scanning Transmission Electron Microscope (STEM) was utilized in order to study the microstructure (precipitated phases) in Inconel718. Objective was to understand the effect o f heat treatment on these precipitated phases. For this purpose material was subjected to: aging cycle and annealing treatment Details of heat treatment are given in Table 4.

IOP Conf. Series: Materials Science and Engineering **146** (2016) 012051 doi:10.1088/1757-899X/146/1/012051

Table 4. Aging cycle and annealing treatment for Inconel718 alloy

Aging Cycle	Annealing Treatment
Solution Treatment at 980°C/1hr/air cool	Heat at 960°C for 1 hr.
Heat at 720°C for 8hrs.	
Heat at 620°C for 8hrs.	Air cool
Air cool	

Figure 4 shows γ′ and γ″ precipitates in peak aged inconel718. These precipitates are responsible for imparting strength to the super alloy. Ni_3Nb (γ″) was identified as the dominant hardening phase. Both γ′ and γ″ are metastable in nature. Figure 5 shows the Selected Area Diffraction (SAD) patterns of γ″ precipitates obtained using the diffraction mode. Habit plane of γ′ and γ″ is {100} at [$\bar{1}$00] orientation. It was observed that γ″ precipitates grew perpendicular to [$\bar{1}$00]. Fully coherent relationship was observed γ″ phase and austenitic matrix. Ellipsoidal disc shaped γ″ phase was observed. Theses precipitates existed on (100), (010) and (001) planes at [$\bar{1}$00] orientation.

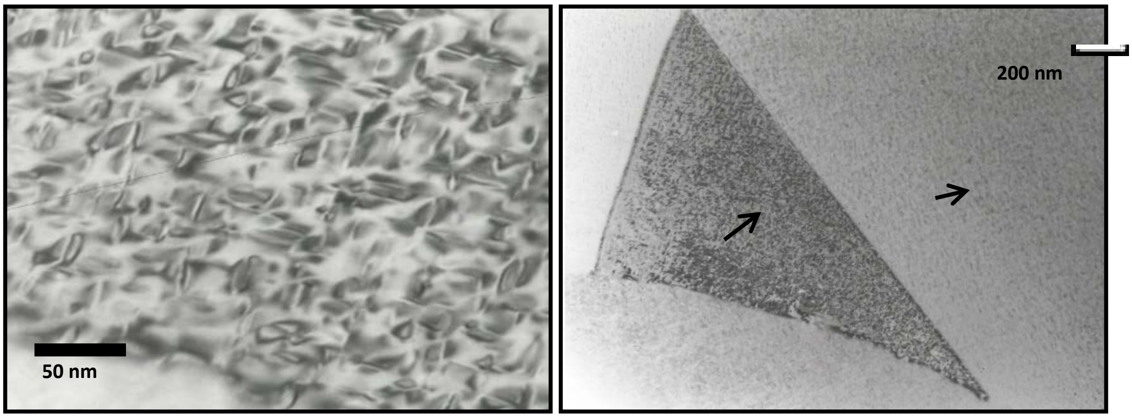

Figure 4. STEM micrographs depicting γ′ and γ″ in peak aged inconel718 alloy

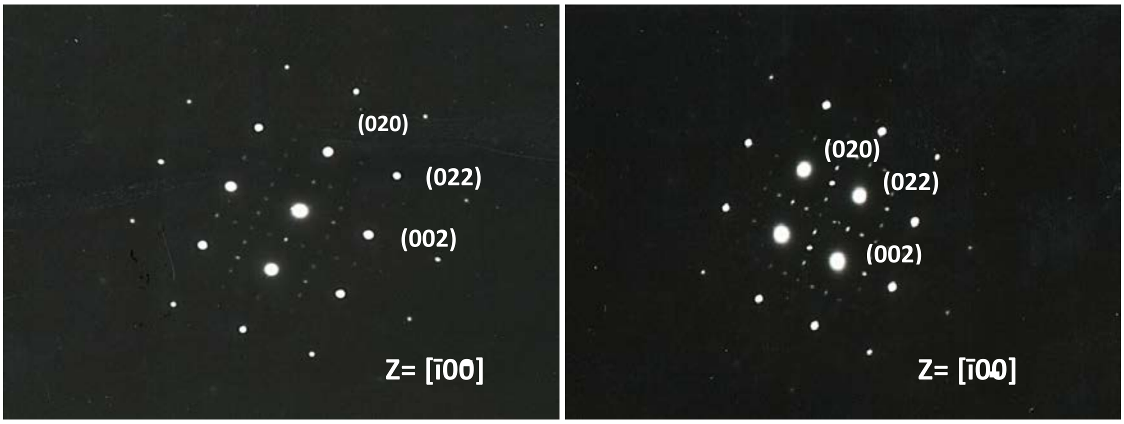

Figure 5. SAD pattern of γ″ precipitates

Figure 6 shows STEM micrographs of annealed Inconel718 alloy. In annealed condition needle like δ phase was observed. This δ phase resulted due to transformation of metastable γ″ to stable δ. Yun Z et al. reported that δ equilibrium phase nucleates at γ″ at temperature< 1173K [10]. δ can also precipitate directly in γ matrix at higher aging temperatures. Cubic/blocky niobium carbides were also observed in annealed samples. Figure 7 shows selected area diffraction pattern of annealed sample. Zone axis was calculated as Z: [2$\bar{1}$1].

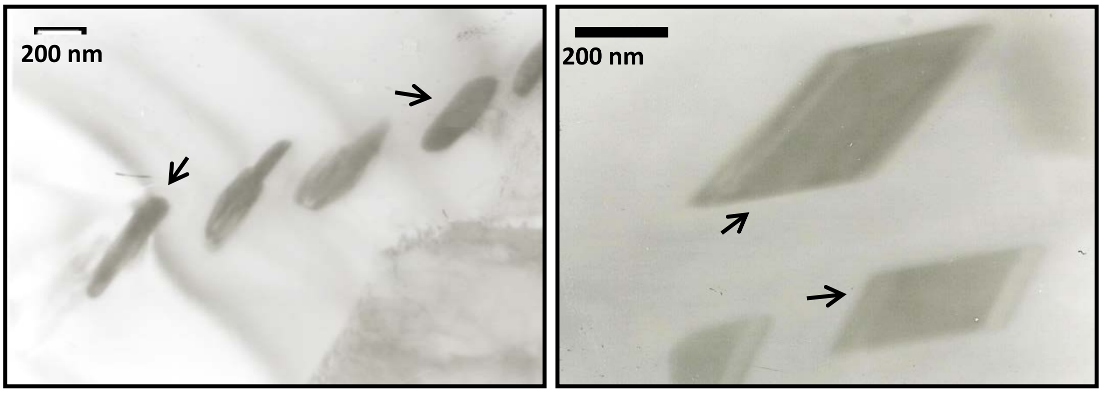

Figure 6. STEM micrographs depicting δ phase (left) and blocky NbC (right) in annealed state

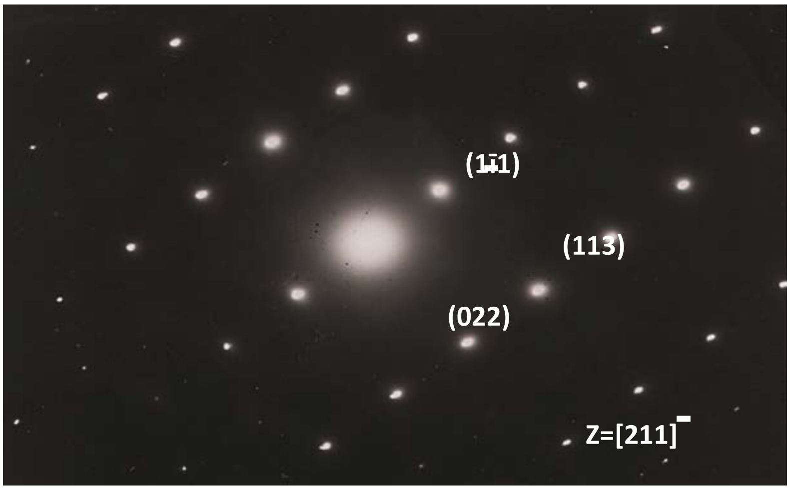

Figure 7. SAD pattern of annealed inconel718 samples

4. Conclusion

i. Ti content has a profound effect on high temperature rupture ductility of Inconel718.
ii. (Al+Ti+Nb) at% was found to be 5.98. However for typical Inconel718 with 1 wt. % Ti, (Al+Ti+Nb)at% = 5.5. Thus high temperature stress rupture properties of Inconel718 sensitively depend on (Al + Ti + Nb) at%.
iii. Ti content above 1% leads to formation of TiC in large quantities. 5-6 µm primary carbides (TiC) were found at grain boundaries in samples subjected to creep testing (649 °C). Hence, presence of MCs (TiC & NbC) in large concentrations at grain boundaries deteriorated creep properties.
iv. Fractographs of fractured samples during creep testing (649 °C/ 690MPA) revealed that large primary microvoids nucleated at (Nb, Ti) C.
v. γ′ + γ″ were observed to be primary strengthening phases in as-received Inconel718. Habit plane of γ′ and γ″ is {100} at [$\bar{1}$00] orientation. γ″ precipitates grow perpendicular to [$\bar{1}$00]. Upon annealing metastable γ″ transformed to stable acicular δ phase (Ni_3Nb).
vi. Fully coherent relationship between γ″ precipitates and matrix was found. Ellipsoidal disc shaped γ″ precipitates lie on (100), (010) & (001) planes at [$\bar{1}$00] orientation.

5. References

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