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NAVAL AIR WARFARE CENTER AIRCRAFT DIVISION PATUXENT RIVER, MARYLAND

# TECHNICAL INFORMATION MEMORANDUM



REPORT NO: NAWCADPAX/TIM-2016/216

## THERMAL FATIGUE OF INCONEL ALLOY DA718

by

E. U. Lee M. Stanley B. Pregger

27 October 2016

Approved for public release.

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#### DEPARTMENT OF THE NAVY NAVAL AIR WARFARE CENTER AIRCRAFT DIVISION PATUXENT RIVER, MARYLAND

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#### SUMMARY

An Inconel alloy DA718 specimens were subjected to thermal fatigue cycling between the predetermined maximum temperature  $T_{max}$  and the minimum temperature  $T_{min}$ , or with addition of holding at the both temperatures. The  $T_{max}$  ranged from 1200 to 1550°F, and the  $T_{min}$  was 600°F. During the thermal fatigue cycling, the specimen was constrained to prevent any longitudinal expansion or shrinkage in a MTS machine. The induced axial stress, diametral strain, and specimen temperature were continuously measured. The test results indicated that

- The thermally induced minimum axial stress became less compressive with increasing thermal cycle, higher  $T_{max}$ , and longer holding period.
- The diametral strain at the specimen mid-length changed cyclically during thermal fatigue cycling. The maximum was constant initially and decreased later. This was noticed earlier for higher temperature and longer holding time.
- During the thermal fatigue cycling, intragrain  $\gamma'$  and  $\gamma''$  precipitates were coarsened,  $\gamma''$  transformed to  $\delta$  phase, and  $\gamma''$ -depleted zones were formed near grain boundaries. Those zones were prone to intergranular cracking.

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#### INTRODUCTION

#### BACKGROUND

Structural components experience various operating conditions, often involving complex combination of cyclic temperature, cyclic mechanical stress, and varying environments. Especially, some of them are subjected to a variety of thermal and thermomechanical loads. If the stresses in a component develop under thermal cycling without external loading, the term thermal fatigue or thermal stress fatigue is used. On the other hand, thermal fatigue can also develop even under conditions of uniform temperature, caused by internal constraints such as different grain orientations or anisotropy of the thermal expansion coefficient of different metallurgical constituents. Internal stress and strain can be of sufficiently high magnitude to cause distortion, cracking and/or surface irregularities in the material. Consequently, thermal cycling results in damage and deterioration of the microstructure. By following the changes in microstructure, and by observing the changes in distribution of important microconstituents with increasing thermal damage, it would be possible to gain some knowledge of the mechanism of thermal fatigue.

Though thermal fatigue failures are by no means a new problem, the trends in modern technology to higher temperatures and cyclic operations have increased their occurrence and damaging effects. Many examples have been discussed in the literature, for example, aircraft engines, power generating plants, petroleum refineries, and nuclear reactors. Considering the importance, there is little fundamental information available on thermal fatigue of ductile materials.

#### **PURPOSE**

The purpose of this study is to clarify the effects of the temperature and number of thermal fatigue cycle, and the concurrent microstructural change on the thermal fatigue behavior of a ductile material, and establish its controlling mechanism.

#### **METHODS**

#### MATERIAL

A commercial Inconel alloy 718 is a precipitation hardenable alloy designed to have high yield, tensile and creep-rupture properties. There are three versions of wrought Inconel alloy 718 used in the production of gas turbine engine components (reference 1). "Standard Processed" alloy 718 is used for noncritical or difficult to make shapes and has an average grain size of ASTM 4-6. "High Strength Processed" alloy 718 is used for more highly stressed components with less complex configurations and has an average grain size of ASTM 8. The third version, "Direct Age Processed" alloy 718 or DA718 achieves the highest tensile at a further expense in shape making capability. This material has an average grain size of ASTM 10, and used in disc application where high tensile and fatigue strength are required. The DA718 shows significant improvements in tensile strength and low cycle fatigue properties, but exhibits low stress rupture

life in the low stress-high temperature regime. For compressor and turbine disc application, this material meets the required improvement and offers a low cost alternative to powder metallurgy Rene'95. However, its thermal fatigue resistance remains to be fully clarified.

Its nominal chemical composition is shown in Table B-1. In the fully heat treated condition, DA718 consists of a  $\gamma$  matrix,  $\gamma'$ ,  $\gamma''$ ,  $\delta$  precipitate phases, and small amounts of NbC carbides and TiN nitrides. The major strengthening precipitate in DA718 is the Ni<sub>3</sub>Nb  $\gamma$ '' phase, which is coherent and has a DO<sub>22</sub> crystal structure (reference 2). This precipitate is disc-shaped and has a diameter of 20-40  $\mu$ m after conventional aging treatments. The  $\gamma$ '' strengthening is due to coherency strains that arise during precipitation. A smaller amount of Ni<sub>3</sub>(Al,Ti)  $\gamma$ ' phase (L1<sub>2</sub> structure) is present in the alloy, and this precipitate forms as a spherical shaped particle. The  $\gamma$ ' phase begins to solution between 1550 and 1600°F, while the solvus of the  $\gamma$ ' phase is somewhat lower. Both the  $\gamma'$  and  $\gamma''$  phases are metastable, whereas the stable phase is Ni<sub>3</sub>Nb  $\delta$ , which is incoherent and has an orthorhombic crystal structure. This phase forms as intragranular laths along  $\{111\}$ , or at grain boundaries. Precipitation of the  $\delta$  phase is usually more pronounced in the 1700-1750°F range, and the solvus is typically near 1800°F. The  $\delta$  phase plays an important role in the control of grain size, since its presence can inhibit grain coarsening during processing and heat treatment. In this study, the DA718 was chosen as the specimen material, and its scanning electron micrograph is shown in Figure A-1. This figure indicates  $\delta$  phase plates along grain boundaries and within grains, and tiny intragrain  $\gamma$ ' and  $\gamma$ '' precipitates in  $\gamma$  matrix.

#### **SPECIMEN**

The employment of an hourglass specimen in thermal fatigue tests would be of vital benefit in improving the geometry stability. A uniform longitudinal temperature distribution is not required; it is only necessary that the maximum temperature occurs in the minimum cross section. The use of induction heating can produce a uniform radial temperature. Only the diametral strain and temperature need to be recorded as a function of thermal load in order to compute the actual strain range in the minimum cross section. Therefore, the hourglass specimen was selected as the test specimen in this study.

Hourglass specimens were machined from a 1.5 in. diameter round bar of DA718 in Lorientation. The length was 6 in., the minimum diameter 0.25 in., the blending fillet radius 2.25 in., and the grip section diameter 0.5 in.

#### TEST APPARATUS

The thermal fatigue testing apparatus consists of an induction heating coil, a tubular ring diffuser of compressed air with several holes, both of which surround a specimen, and a diametral extensometer at the specimen mid-length in a MTS machine, Figure A-2.

Its essential feature is the imposition of a complete longitudinal constraint on the test specimen with a couple of locked hydraulic grips in a MTS machine. To develop a cyclic thermal stress in the test specimen, the specimen temperature is cycled by programmed induction heating and compressed-air cooling. A solenoid-operated valve is used to supply compressed-air to the tubular ring diffuser during the cooling phase of the cycle.

The longitudinal or axial load or stress, induced by the expansion and contraction of the specimen during thermal cycling, is measured by a load-cell above the specimen. The specimen temperature during the testing is measured with a chromel-alumel thermocouple, spot-welded to the mid-length of the specimen. The thermal strain, induced by the expansion and contraction of the specimen, is measured with a diametral extensometer at the mid-length.

## THERMAL FATIGUE CYCLING

The specimen temperature was cycled between the predetermined maximum temperature of the cycle  $T_{max}$  and the minimum temperature of the cycle  $T_{min}$ , or with addition of holding at the both temperatures. The former was defined as Simple Thermal Cycling, and the latter as Thermal and Holding Cycling in this study. The Simple Thermal Cycling was broken down into two components: (1) heat and (2) cool. The Thermal and Holding Cycling was broken down into four components: (1) heat, (2) hold at  $T_{max}$ , (3) cool, and (4) hold at  $T_{min}$ . The  $T_{max}$  ranged from 1200 to 1550°F, and the  $T_{min}$  was 600°F. The time required for each component was longer for higher  $T_{max}$ , taking account of the longer time of induction heating and compressed air cooling. The schemes of the thermal fatigue cycling are shown in Figures A-3(a) and (b), respectively.

During the thermal fatigue cycling, the changing axial (or longitudinal) load in the specimen was measured with a load-cell in the MTS machine continuously. The minimum diameter of the specimen at the mid-length was measured with an extensometer continuously. The measured axial load and diameter of the specimen were converted to the axial stress and diametral strain, and saved in the attached computer.

## MICROGRAPHY AND FRACTOGRAPHY

The microstructure of the polished and etched specimen cross-section and the morphology of the specimen fracture surface were examined with a JEOL JSM-6460LV scanning electron microscope, operated at an accelerating voltage of 20 kV.

## EXPERIMENTAL RESULTS

#### VARIATION OF AXIAL STRESS DURING SIMPLE THERMAL CYCLING

The axial or longitudinal stress, induced by thermal cycling, was found to be compressive and changing cyclically. The minimum axial stress of each thermal cycle increased with the increasing number of thermal cycle and increasing maximum temperature, as shown in Figure A-4. In other words, the thermally induced stress became less compressive with increasing number of thermal cycle and increasing maximum temperature applied.

### VARIATION OF DIAMETRAL STRAIN DURING SIMPLE THERMAL CYCLING

During the thermal fatigue cycling, the specimen was bulged cyclically at the heated center of the constrained specimen, because of compressive yielding and creep. Correspondingly, the diametral strain changed cyclically. The maximum diametral strain at the mid-length was around 0.01 in./in. and changed little during the thermal cycling for the maximum temperatures below 1400°F, but it decreased with increasing number of thermal cycle at and above 1400°F, as shown in Figure A-5. The decrease in the maximum diametral strain was greater and occurred earlier for the higher maximum temperature of the thermal cycle.

#### VARIATION OF AXIAL STRESS DURING THERMAL AND HOLDING CYCLE

The features of the minimum axial stress variation during thermal and holding cycling are similar to those for the simple thermal cycling, except the lower minimum axial stress or less compression, as shown in Figure A-6.

#### VARIATION OF DIAMETRAL STRAIN DURING THERMAL AND HOLDING CYCLE

During the thermal and holding cycling, the maximum diametral strain was about 0.01 in./in. for an initial period and began to decrease at or above 1300°F of the maximum cycle temperature. In the case of maximum cycle temperature 1300°F, the period was 200 cycles, but it was shorter for higher maximum cycle temperatures. On the other hand, for the maximum cycle temperature 1200°F, the maximum diametral strain was 0.0028 in./in. up to 155 cycles and then gradually decreased. Those variations of the maximum diametral strain with number of thermal and holding cycle are shown in Figure A-7.

## COMPARISON OF MINIMUM AXIAL STRESSES FOR SIMPLE THERMAL CYCLING AND THERMAL AND HOLDING CYCLING

The minimum axial stresses  $(\sigma_z)_{min}$  for the simple thermal cycling and the thermal and holding cycling are compared in Figure A-8. At the five different maximum cycle temperatures  $T_{max}$ , 1200, 1300, 1350, 1400, and 1550°F, and throughout the applied numbers of thermal cycle, the minimum axial stress is less compressive for the thermal and holding cycling than for the simple thermal cycling.

#### MICROGRAPHY

The microstructures of the specimens, subjected to simple thermal cycling and thermal and holding cycling, are shown in Figures A-9 and A-10, respectively. Figure A-9(a) depicts an intergranular fracture of a specimen, which was undergone to simple thermal cycling of 600-1350°F. In Figure A-9(b), it is noticeable that  $\gamma$ '' precipitates were depleted and cracks appeared in the zones along grain boundaries during the simple thermal cycling of 600-1400°F. In Figure A-10, it is observable that the initially equi-axed grains (Figure A-1) were elongated and bowed,

and the intragrain and intergranular  $\delta$  phase rods were bent or bowed during the thermal and holding cycling of 600-1500°F.

#### FRACTOGRAPHY

Typical SEM fractographs for the specimens, subjected to simple thermal cycling and thermal and holding cycling, are shown in Figures A-11 and A-12, respectively. They indicate:

- A crack was initiated on the specimen surface, and grew inward, creating a slow crack growth area and an overload fracture area.
- The slow crack growth area has beach-marks, containing fatigue-facets with striations, secondary cracks along some striations and cracks along some facet-boundaries.
- In the overload fracture area, equi-axed dimples are noticeable. Near the boundary between the slow crack growth area and overload fracture area, a mixture of dimples and fatigue facets with striations is seen.
- The fractogaphic features are similar to those for the mechanical fatigue at room temperature.

#### DISCUSSION

#### STRESS AND STRAIN UNDER THERMAL FATIGUE CYCLING

For an axial stress in an hourglass specimen, induced by thermal fatigue cycling, the radial or diametral strain can be expressed as (reference 3)

$$\varepsilon_{\rm r} = \alpha T - \mu \left( \sigma_{\rm z} / E \right) - \left( \varepsilon_{\rm z} / 2 \right) \tag{1}$$

where

- $\varepsilon_r$ : radial or diametral strain
- $\alpha$ : coefficient of thermal expansion
- T: temperature
- μ: Poisson's ratio
- $\sigma_z$ : axial stress
- E: Young's modulus
- $\varepsilon_z$ : axial strain

In this study, the specimen was constrained with a pair of hydraulic grips, and so there was no axial strain or  $\varepsilon_z = 0$ . Therefore, equation (1) can be simplified as

$$\varepsilon_{\rm r} = \alpha T - \mu(\sigma_{\rm z}/E) \tag{2}$$

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On the one hand, the radial or diametral strain  $\varepsilon_r$  can be measured with a diametral extensometer. That strain for the minimum radius R of the specimen is

$$\varepsilon_{\rm r} = \Delta D/2R \tag{3}$$

where

ΔD: diametral displacementR: minimum radius of specimen

The axial stress  $\sigma_z$  can be measured with a load-cell above the specimen in the MTS machine. From Equations (2) and (3),

$$\Delta D/2R = \alpha T - \mu(\sigma_z/E) \tag{4}$$

The  $\Delta D/2R$  and  $\sigma_z$  were measurable during the thermal fatigue cycling in this study. The validity of this equation was demonstrated by the measured  $\Delta D/2R$  and  $\sigma_z$  values, and the reported  $\alpha$ ,  $\mu$  and E values (references 4 and 5) for the  $T_{max} = 1200$  and  $1400^{\circ}F$ , as shown in Table B-2.

#### VARIATION OF AXIAL STRESS AND DIAMETRAL STRAIN DURING THERMAL FATIGUE CYCLING

The measured minimum axial stress  $(\sigma_z)_{min}$ , induced by simple thermal cycling or thermal and holding cycling was compressive. Its absolute value decreased (or became less compressive) with increasing number of cycle N and increasing maximum temperature  $T_{max}$ , Figures A-4 and A-6. This behavior is similar to the stress relaxation of the specimen material, observed by some investigators during their studies on thermal and thermomechanical fatigue of structural alloys (reference 6). Such a stress relaxation is strongly temperature-dependent, and it occurs readily during the holding interval at elevated temperatures (reference 7).

Balandin (reference 8) reported that the increase of the maximum test temperature reduced the number of cycles before cracks develop. This was explained mainly by the increased thermal stresses on account of the larger temperature gradient. A considerable effect on the reduced resistance to thermal fatigue can be exerted also by a general lowering of the mechanical properties with increasing temperature, by accelerated creep and an increased coefficient of expansion.

The maximum diametral strain  $(\varepsilon_r)_{max}$  was measured to be nearly constant below 1550°F of the maximum temperature of the cycle  $T_{max}$ , whereas it decreased with increasing time or number of thermal cycle at  $T_{max} = 1550$ °F under simple thermal cycling, Figure A-5. On the one hand, it decreased with number of cycle at 1300°F or higher  $T_{max}$  under thermal and holding cycling, Figure A-7.

## <u>COMPARISON OF MINIMUM AXIAL STRESSES FOR SIMPLE THERMAL CYCLING</u> <u>AND THERMAL AND HOLDING CYCLING</u>

The minimum axial stresses  $(\sigma_z)_{min}$  for the simple thermal cycling and the thermal and holding cycling are compared in Figure A-8. At the five different maximum cycle temperatures  $T_{max}$ , 1200, 1300, 1350, 1400, and 1550°F, and throughout the applied numbers of thermal cycle, the minimum axial stress is less compressive for the thermal and holding cycling than for the simple thermal cycling. This behavior is attributable to the holding at the maximum and minimum temperatures, during which presumably more microstructural degradation can occur.

#### MICROSTRUCTURAL CHANGE DURING THERMAL FATIGUE CYCLING

The microstructure was observed to change more with longer cycling and holding time and higher heating temperature, Figures A-9(b) and A-10. The microstructural change consisted of coarsening of intragrain precipitates  $\gamma'$  and  $\gamma''$ , transformation of  $\gamma''$  to  $\delta$  phase, and formation of  $\gamma''$ -depleted zone adjacent to grain boundaries. Such a microstructural change in alloy 718 was also observed by others (references 9-13). The zone without strengthening precipitate  $\gamma''$  is weaker than the grain interior and unable to withstand high stresses. As the result, the zone is prone to cracking or intergranular cracking. The observed intergranular fracture, shown in Figure A-9(a), is believed to be attributed to the extension and interconnection of the intergranular cracking. Glenny (references 14 and 15) and Franklin (reference 16) conducted studies on thermally fatigued Nimonics, and reported that above a maximum cycle temperature of 800°C intergranular fracture predominated but below this temperature transgranular fracture was usual.

On the basis of the above discussion, it is clear that the variations of  $(\sigma_z)_{min}$  and maximum diametral strain $(\varepsilon_r)_{max}$  are attributed to the microstructural degradation during the thermal cycling and thermal and holding cycling. Furthermore, the mechanism of thermal fatigue failure must be the intergranular cracking, arising from the microstructural degradation of the specimen material

The elongated and bowed grains and bent  $\delta$  rods, shown in Figure A-10, evidence that the specimens crept under compression during the thermal and holding cycling of the constrained specimens.

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#### CONCLUSIONS

- The variation of the minimum axial stress and maximum diametral strain is attributed to the microstructural degradation during the simple thermal cycling and thermal and holding cycling.
- The mechanism of thermal fatigue failure is intergranular cracking, arising from the microstructural degradation of the specimen material.
- The microstructural degradation consists of growth of  $\gamma$ ' and  $\gamma$ '' precipitates, phase transformation of  $\gamma$ '' to  $\delta$ , and formation of  $\gamma$ ''-depleted zones along grain boundaries, which are prone to intergranular cracking.

## RECOMMENDATION

• Investigate the thermomechanical fatigue behavior of DA718.

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## APPENDIX A LIST OF FIGURES

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Fiugre A-1: Scanning Electron Micrograph of Super Ally Inconel 718



Figure A-2: Apparatus for Thermal Fatigue Testing



Figure A-3(a): Simple Thermal Cycle



Figure A-3(b): Thermal and Holding Cycle



Figure A-4: Variation of Minimum Axial Stress with Number of Simple Thermal Cycle



Figure A-5: Variation of Maximum Diametral Strain with Number of Simple Thermal Cycle



Figure A-6: Variation of Minimum Axial Stress with Number of Thermal and Holding Cycle



Figure A-7: Variation of Maximum Diametral Strain with Number of Thermal and Holding Cycle



Figure A-8: Variation of Minimum Axial Stress under Simple Thermal Cycling and Thermal and Holding Cycling (\*1200: Simple Thermal Cycling at Max. Temp 1200°F; 1200X: Thermal and Holding cycle under Max. Temp 1200°F)



Figure A-9(a): Intergranual Fracture in Specimen, Subjected to Simple Thermal Cycling of 600-1350°F



Thermal A-9(b): Intergranual Cracking in Specimen, Subjected to Simple Thermal Cycling of 600-1400°F



Figure A-10: Elongated Grains, and Bowed or Bent  $\delta$  Rods within and along Grain Boundaries in Specimen, Subjected to Thermal and Holding Cycling at 600-1500°F



Figure A-11: SEM Fractographs of Specimen, Subjected to Simple Thermal Cycling of 600-1550°F



Figure A-12: SEM Fractographs of Specimen, Subjected to Thermal and Holding Cycling of 600-1400°F

#### APPENDIX B TABLES

## Table B-1: Chemical Composition (wt %) of Super Alloy Inconel 718

Al	0.2 - 0.8	Mo	2.8 - 3.3
В	0.006 max	Ni	50 - 55
С	0.08 max	Nb	4.75 - 5.5
Cr	17 -21	Р	0.015 max
Co	1 max	Si	0.35 max
Cu	0.3 max	S	0.015 max
Fe	Balance	Ti	0.65 - 1.15
Mn	0.35 max		

Table B-2: Validation of Thermal Stress and Strain Relationship  $\Delta D/2R = \alpha T - \mu(\sigma_z/E)$ 

Т	α	αΤ	E	$\sigma_{z}$	μ	$\mu\sigma_z$	μσ <sub>z</sub> /Ε	[αT - (μσ <sub>z</sub> /E)]		$\Delta D/2R$
		<u>(°</u> F)	<u>(10<sup>-6</sup>/<sup>°</sup>F</u> )	<u>(10<sup>-2</sup>) (psi</u>	<u>x 10<sup>3</sup>)</u>	<u>(psi</u> )	<u>(psi</u> )	<u>(10<sup>-3</sup>)</u>		
1200	8.4	1.008	23.7	-400	0.283	-113.2	-4.776	0.0149	≅	0.01303
1400	8.9	1.246	22.3	-120	0.306	-36.7	-1.646	0.0141	$\cong$	0.01216

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