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It is certified that the work contained in the thesis titled ***"Tensile and Low Cycle Fatigue Behaviour of Superalloy Inconel 617"*** by ***"Ch. Visweswara Rao"*** has been carried out under my supervision and this work has not been submitted elsewhere for a degree.

It is further certified that the student has fulfilled all the requirements of Comprehensive, Candidacy and SOTA for the award of PhD degree.

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DECLARATION BY THE CANDIDATE

I, ***"Ch. Visweswara Rao"***, certify that the work embodied in this thesis is my own bonafide work and carried out by me under the supervision of ***"Prof. N. C. Santhi Srinivas"*** from ***"July 2015"*** to ***"December 2020"***, at the ***"Department of Metallurgical Engineering"***, Indian Institute of Technology (BHU), Varanasi. The matter embodied in this thesis has not been submitted for the award of any other degree/diploma. I declare that I have faithfully acknowledged and given credits to the research workers wherever their works have been cited in my work in this thesis. I further declare that I have not wilfully copied any other's work, paragraphs, text, data, results, *etc*., reported in journals, books, magazines, reports dissertations, theses, *etc*., or available at websites and have not included them in this thesis and have not cited as my own work.

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It is certified that the above statement made by the student is correct to the best of our knowledge.

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Ch. Visweswara Rao

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Symbol	Description
c	fatigue ductility exponent
b	fatigue strength exponent
e	engineering strain
e_{pf}	plastic strain to fracture/ elongation up to fracture
e_{pn}	necking plastic strain/ elongation up after necking
e_{pu}	uniform plastic strain/ elongation up to ultimate tensile strength
ε	true strain
ε_{c}	critical plastic strain for the onset of serrations
$\dot{\varepsilon}$	strain rate
ε_{o}	pre-strain existing in the material
$\Delta\varepsilon_{\mathrm{e}}$	elastic strain range
$\Delta\varepsilon_{\mathrm{e}}/2$	elastic strain amplitude
$\Delta\varepsilon_{\mathrm{p}}$	plastic strain range
$\Delta\varepsilon_{\mathrm{p}}/2$	plastic strain amplitude
$\Delta\varepsilon_{\mathrm{t}}$	total strain range
$\Delta\varepsilon_{\mathrm{t}}/2$	plastic strain amplitude
$\Delta\mathrm{H}$	degree of hardening;
ε_f'	fatigue ductility coefficient
K	strength coefficient
K_1	additional constant defined in Ludwigson equation
K'	cyclic strength coefficient
m	strain rate sensitivity exponent
n	strain hardening exponent
n_1	additional constant defined in Ludwigson equation
n'	cyclic strain hardening exponent
N	number of cycles
N_{i}	number of cycles to crack initiation

N_f	number of cycles to failure
Q	activation energy
R	universal gas constant
S_{UTS}	ultimate tensile strength
S_{YS}	yield strength
σ	true stress
σ_a	stress amplitude
σ_b	back stress
σ_f	friction stress
σ_T	tensile stress amplitude at half-life
σ_s	saturation stress
σ'_f	fatigue strength coefficient
θ	work hardening rate
ΔW_e	elastic strain energy per cycle
ΔW_p	average plastic strain energy per cycle
ΔW_t	total plastic strain energy per cycle

PREFACE

Inconel 617 is a nickel based superalloy which contains Cr, Co, Mo, Al and Ti as alloying elements. It is widely used in high temperature applications because of its superior creep resistance and improved stability of microstructure for long exposures at elevated temperatures. A protective layer of Cr and Al oxides forms on the surface of the alloy at elevated temperature which enhances its oxidation resistance. This tungsten free alloy is lighter and cost competitive as compared to other nickel based alloys with tungsten. It is primarily strengthened by precipitation of fine homogeneously dispersed γ' phase in the γ matrix and from the precipitation of M_6C and $M_{23}C_6$ carbides both at grain boundaries and in the grains. Inconel 617, among Ni base super alloys, is the prime candidate material for tubing in super heater, re-heater and steam generator components due to its excellent creep strength and resistance to oxidation. It has been widely used also in other high temperature applications such as intermediate heat exchanger (IHX) in very high temperature gas cooled reactors (VHTR), combustor liners, transition ducting and exhaust system components of aircraft and land based gas turbine engines, catalyst-grid supports in the production of nitric acid, heat-treating baskets and reduction boats in the refining of molybdenum.

Advanced ultra super critical (A-USC) coal fired power plants using steam at high temperatures up to 780^{o}C and pressure of 35 MPa have been developed, to increase efficiency and reduce CO_2 emissions. These plants require high performance alloys to resist such high temperatures and pressures. Nickel based super alloys with high creep strength and corrosion resistance are candidate materials for the hottest boiler and turbine sections. Inconel 617 is the prime material of usage among these alloys. Application of Inconel 617 alloy in boilers of A-USC power plants requires understanding of the overall mechanical properties and related deformation mechanisms over wide range of temperatures under tensile, creep and cyclic loading. The primary mode of failure of components made of Inconel 617 is due to cyclic loading (fatigue) resulting during start-up and shut down operations. Temperature induced cyclic strains of different magnitude are also produced by thermal stresses generated during such operations. Thus, understanding of the low cycle fatigue behaviour and related deformation mechanisms at room temperature as well as at elevated temperatures is necessary. Inconel 617 alloy when used as combustion lining in combustion chamber of gas turbines is exposed to severe high temperature and corrosive environments which may affect its performance. Thermal barrier coatings (TBC) can improve the life of these alloys at high temperatures, whereas salt environment is detrimental to the life of these alloys. However, only limited investigations have been carried out on Inconel 617 alloy to evaluate the effect of temperature, microstructure and environment on deformation and fracture behaviour under tensile and fatigue loading. The present work aims to investigate the tensile and low cycle fatigue behaviour of the Inconel 617 alloy under various conditions in detail.

Chapter 1 discusses brief introduction about the alloy design and development of nickel-based superalloys in general and Inconel 617 in particular along with the physical metallurgy and intended applications of these alloys. Literature related to microstructural characterization and mechanical properties of Inconel 617 alloy subjected to tensile and cyclic loading till date is also reviewed. The need and importance of the present investigation is emphasized along with the objectives of present investigation.

Chapter 2 presents details of material and experiments carried out in the present investigation. Inconel 617 alloy was procured as forged rod of 14 mm diameter and was solution annealed at 1175^{O}C for 40 minutes and quenched in water. Tensile behaviour of the Inconel 617 alloy in solution annealed condition was investigated in the temperature range from RT to 900^{o}C at different strain rates from $5x10^{-4}$ s^{-1} to $1x10^{-2}$ s^{-1} to establish the process of dynamic strain aging (DSA) and examine its effect on deformation and fracture behaviour. Work hardening behaviour of the Inconel 617 alloy was analyzed in different conditions (solution annealed, aged, and cold worked) at room temperature and 700^{o}C after conducting tensile testing at a strain rate of $5x10^{-3}$ s^{-1}.

Low cycle fatigue (LCF) testing was performed in solution annealed condition at room temperature, 750^{o}C and 850^{o}C in air, under fully reversed axial strain-controlled mode at constant strain rate of $5x10^{-3}$ s^{-1}. Symmetrical triangular wave form and total strain amplitude ranging from ±0.20% to ±0.50% were used at the three temperatures to study fatigue behaviour. LCF tests were also conducted on samples subjected to oxidation at 850^{o}C with and without salt coating for durations up to 1000 h. LCF tests on these samples were conducted at strain amplitude of ±0.25%, at constant strain rate ($\dot{\varepsilon}$) of $5x10^{-3}$ s^{-1}. Fracture surfaces of the tensile and fatigue tested samples were examined by scanning electron microscope. Deformation behaviour was analyzed using transmission electron microscopy. Thermal barrier coating (TBC) of Yttria (8%) stabilised Zirconia (YSZ) was applied on fatigue samples using air plasma spray (ASP) coating process. The samples were initially grit blasted, and then were applied with a bond coat of NiCrAlY of ≈40 µm thick and coating of YSZ of ≈80µm thick. Effect of strain amplitude on the fatigue life of the TBC coated samples was studied by conducting LCF testing at strain amplitudes from ±0.20% to ±0.50% at 850^{o}C.

Chapter 3 deals with the tensile deformation behaviour of Inconel 617 alloy and establishes the DSA regime for the alloy. Serrations were observed in the temperature range 300^{o}C-700^{o}C at all the three strain rates studied which indicated the occurrence of dynamic strain aging (DSA). The amplitude of serrations increased with increase in the temperature and decrease in the strain rate. Temperature regime of DSA was confirmed to be from 300^{o}C to 700^{o}C by the occurrence of plateau in the yield strength, ductility minima and negative strain rate sensitivity in this regime. Activation energies for the serrated flow were found to be 65, 80 and 110 kJ/mol for the three types of serrations, namely B, (A+B) and C respectively. The controlling mechanism of the DSA was found to be diffusion of carbon through dislocation cores in the lower temperature range and diffusion of substitutional elements Cr and Mo in the higher temperature range. TEM studies revealed increase in the number of slip bands as well as interaction of dislocations with solute atoms with increase in the temperature up to 700^{o}C. Precipitation of carbides was observed at 700^{o}C, at the ductility minima. Above this temperature, increase in precipitate size and sub-grain formation was observed. SEM examination of the fracture surfaces revealed ductile fracture with dimples at room temperature and 400^{o}C-600^{o}C, where distinct serrations were observed in the flow curve. At 700^{o}C, at ductility minima, there was mixed mode of fracture with dimples, facets and intergranular cracks associated with some grains boundaries. At 800^{o}C and 900^{o}C, there was completely ductile fracture with large and deep dimples.

Chapter 4 describes the effect of microstructure on work hardening behaviour of Inconel 617 alloy at RT and 700^{o}C in different conditions (as received, solution annealed, aged, and cold worked) of the alloy. Tensile testing was conducted at room temperature and 700^{o}C (operating temperature of boilers in A-USC power plants), at a strain rate of 5x10^{-3} s^{-1}. True stress-true strain curves of the alloy displayed concave slope upward similar to those of other austenitic super alloys. Five different flow relationships were examined for all these conditions of the alloy to characterize its work hardening behaviour. Ludwigson relationship was found to be the best to describe flow behaviour of Inconel 617 alloy. Three different stages were observed in the plots of work hardening rate as a function of true stress. TEM study revealed traces of slip bands at room temperature and formation of carbide precipitates, micro twins and their interaction with dislocations at 700^{o}C, for all the conditions, suggesting deformation by slip at RT and by twinning at 700^{o}C. Typical ductile fracture with dimples was observed in all the conditions except in the cold worked condition where cleavage facets were seen at both the temperatures.

Chapter 5 presents the effect of temperature on low cycle fatigue (LCF), deformation and fracture behaviour at RT, 750 and 850^{o}C at a strain rate of $5x10^{-3}s^{-1}$. Symmetrical triangular wave form and total strain amplitude ranging from ±0.20% to ±0.50% were applied at the three temperatures to study fatigue behaviour. The cyclic stress response and strain life relationship were analysed at these temperatures. While there was continuous cyclic hardening at room temperature at lower strain amplitudes (±0.20% and ±0.25%), cyclic softening was observed after initial cyclic hardening for 100 cycles at higher strain amplitudes (±0.42% and ±0.50%) with a transition at ±0.375% strain amplitude. Continuous cyclic hardening was observed up to peak hardening at 750^{o}C and 850^{o}C irrespective of the strain amplitude. The number of cycles to fracture (fatigue life) decreased with increase in temperature. The degree of hardening increased from RT to 750^{o}C again decreased at 850^{o}C. The degree of cyclic hardening increased with increase in strain amplitude. Non-Masing behaviour was observed at all the three temperatures. TEM studies revealed change in dislocation substructure and formation of precipitates with increase in temperature and strain amplitude. While there was no formation of precipitates, formation of γ' along with $M_{23}C_6$ carbides was observed at 750^{o}C whereas only $M_{23}C_6$ carbides were formed at 850^{o}C. SEM examination of the fracture surfaces revealed increase in inter-striation spacing and extensive branching of cracks at higher strain amplitudes with increase in temperature.

Chapter 6 describes the effect of oxidation and salt coating along with pre exposure up to 1000 hours (cycles of 100 h) on low cycle fatigue behaviour of Inconel 617 alloy at 850^{o}C. Oxidation at 850^{o}C up to 1000 h had no significant effect on fatigue life. A drastic decrease in fatigue life was observed for the salt coated and pre-exposed samples. Elemental mapping by EPMA revealed formation of Cr and Al oxides on the surface for the both oxidised and salt coated samples. These oxide layers were broken under salt environment conditions and the base material was exposed to hot corrosion. The intergranular oxidation depth increased with increase in the duration pre-exposure. Sulphur ingression was observed in the salt coated samples and chromium sulphide (CrS) was found to be detrimental and caused intergranular cracking and reduction in fatigue life.

Chapter 7 discusses the effect of thermal barrier coating on low cycle fatigue behaviour of Inconel 617 alloy at 850^{o}C. Thermal barrier coating of Yttria (8%) stabilised Zirconia (YSZ) was applied on fatigue samples using air plasma spray coating process. At low strain amplitude (±0.2%), the alloy showed improvement in fatigue life whereas at high strain amplitude no improvement in life was observed. The crack initiation started from the base metal surface at low strain amplitude whereas cracks were initiated from TBC coating, under high strain amplitude conditions. Large plastic strain in the samples, tested at high strain amplitude, caused breakage of the surface coatings.

Chapter 8 summarizes the major conclusions of the present investigation and scope for future work.

SUMMARY

Inconel 617 is a nickel base superalloy containing Cr, Co, Mo, Al and Ti as alloying elements. It is primarily strengthened by the M_6C and $M_{23}C_6$ carbides, both present at grain boundaries and within the grains, and the precipitates of fine homogeneously dispersed γ′ phase in the γ matrix. A protective layer of Cr and Al oxides forms on the surface of the alloy at elevated temperature which enhances its oxidation resistance. Inconel 617, among Ni base superalloys, is prime candidate material for tubing in super heater, re-heater and steam generator components, due to its excellent creep strength, resistance to oxidation and improved stability of microstructure after long exposures at elevated temperatures. It has been widely used also in other high temperature applications such as intermediate heat exchanger (IHX) in very high temperature gas cooled reactors (VHTR), combustor liners, transition ducting and exhaust system components of aircraft and land based gas turbine engines. Usage of the Inconel 617 alloy in the above high temperature applications requires understanding of the overall mechanical properties and related deformation mechanisms over a wide range of temperatures under tensile and cyclic loading.

Tensile behaviour of the Inconel 617 alloy in solution annealed condition was investigated in the temperature range from RT to 900^oC at different strain rates from $5x10^{-4}s^{-1}$ to $1x10^{-2}$ s^{-1} to establish the mechanism of dynamic strain aging (DSA) and examine its effect on deformation and fracture behaviour. Serrations were observed in the temperature range of 300^oC to 700^oC, at all the three strain rates studied, which indicated the occurrence of dynamic strain aging (DSA). Temperature regime of DSA was established from 300^oC to 700^oC from the occurrence of plateau in the yield strength, ductility minima and negative strain rate sensitivity, in this regime. The

controlling mechanism of the DSA was found to be diffusion of carbon through dislocation cores in the lower temperature range and diffusion of substitutional elements Cr and Mo in the higher temperature range. TEM examination of the samples revealed, increase in the number of slip bands as well as interaction of dislocations with solute atoms, with increase in the temperature up to 700^{o}C. Precipitation of carbides was observed at 700^{o}C, at the ductility minima. Above this temperature, increase in precipitate size and sub-grain formation was observed. SEM examination of the fracture surfaces revealed ductile fracture with essentially dimples from 400^{o}C to 600^{o}C, where distinct serrations were observed in the flow curve. At 700^{o}C, at ductility minima, there was mixed mode of fracture with features such as dimples, facets and intergranular cracks present at some grains boundaries. At 800^{o}C and 900^{o}C, completely ductile fracture was observed which depicted large and deep dimples.

Work hardening behaviour of the Inconel 617 alloy was analyzed in different conditions (solution annealed, aged, and cold worked) at room temperature and 700^{o}C by conducting tensile tests at a strain rate of $5x10^{-3}$ s^{-1}. True stress-true strain curves of the alloy displayed concave slope upward similar to those of other austenitic superalloys. Ludwigson relationship was found to be the best to describe flow behaviour of Inconel 617 alloy. Three different stages were observed in the plots of work hardening rate as a function of true stress. TEM study revealed traces of slip bands at room temperature and formation of carbide precipitates, micro twins and their interaction with dislocations at 700^{o}C, for all the conditions, suggesting deformation by slip at RT and by twinning at 700^{o}C. Typical ductile fracture with dimples was observed in all the conditions except in the cold worked condition where cleavage facets were seen at both the temperatures.

Low cycle fatigue (LCF) testing was performed in solution annealed condition, at room temperature, 750°C and 850°C in air, under fully reversed axial strain-controlled mode at constant strain rate of $5x10^{-3}$ s^{-1}. Continuous cyclic hardening was observed at all the three temperatures, irrespective of the strain amplitude. The number of cycles to fracture (fatigue life) decreased with increase in temperature. Non-Masing behaviour was observed at all the three temperatures. TEM studies revealed change in dislocation substructure and formation of precipitates with increase in temperature and strain amplitude. While there was no formation of precipitates, formation of γ' along with $M_{23}C_6$ carbides was observed at 750°C whereas only $M_{23}C_6$ carbides were formed at 850°C. SEM examination of the fracture surfaces revealed increase in inter-striation spacing and extensive branching of cracks at higher strain amplitudes with increase in temperature.

LCF tests were also conducted on samples subjected to oxidation at 850°C with and without salt coating for durations up to 1000 h. LCF tests on these samples were conducted at strain amplitude of ±0.25%, at constant strain rate ($\dot{\varepsilon}$) of $5x10^{-3}$ s^{-1}. Oxidation at 850°C up to 1000 h had no significant effect on fatigue life. A drastic decrease in fatigue life was observed for the salt coated and pre-exposed samples. Elemental mapping by EPMA revealed formation of Cr and Al oxides on the surface for the both oxidised and salt coated samples. Sulphur ingression was observed in the salt coated samples and chromium sulphide (CrS) was found to be detrimental and caused intergranular cracking and reduction in fatigue life.

Effect of strain amplitude on fatigue life of the samples of Inconel 617 alloy coated with Thermal Barrier Coating (TBC) of Yttria (8%) stabilised Zirconia (YSZ), was studied by conducting LCF testing at strain amplitudes from ±0.20% to ±0.50% at 850°C. At low strain amplitude (±0.2%), the alloy showed improvement in fatigue life whereas at high strain amplitude, no improvement in life was observed. Fatigue crack initiation started from the base metal surface at low strain amplitude whereas cracks were initiated from TBC coating, under high strain amplitude conditions. Large plastic strain in the samples, tested at high strain amplitude, caused breakage of the surface coatings. The precipitates formed in the coated condition were much less in volume fraction than those tested without TBC coating due to protection of the surface by coating, which reduced the effective temperature of the substrate.