Fractographic investigation of Low Cycle Fatigue behaviour of IN718 coated with NaCl at 550°C

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Abstract. Nickel base super alloys due to their superior properties are used in different parts of gas turbines, in jet engines, and as well as in marine application from 250°C to 650°C. At such high temperature sulphur and vanadium as impurities in fuel oil get oxidised in SO2 and V2O5. Further, sulphur oxide reacts with NaCl to form Na2SO4. These salts cause high temperature corrosion which causes stress corrosion cracking of engine components of marine gas turbine. Therefore, LCF resistance of the material becomes an important consideration in design of turbine. Fatigue samples both coated with NaCl salt and uncoated samples were tested in low cycle fatigue. The fatigue tested samples were analyzed under Scanning Electron Microscope (SEM). Since micrographs analysis is an important tool with the testing of materials to evaluate the various properties of the materials. In present study micrographs analysis helped to a great extent for evaluation the Low cycle fatigue behaviour of Nickel base super alloy INCONAL 718 (IN718) at high temperature which is 550°C.

Keywords: LCF behaviour, IN718, Super alloy, micrographs, hot corrosion.

1. INTRODUCTION

Initial development of superalloys was focused on their high temperature application rather than considering adverse effects of environment conditions during services. Super-alloys are now widely used in various applications at temperatures ranging from 658°C to 1100°C in aggressive atmosphere such as the combustion products of fuel and air, high temperature catalytic reactors [1, 2]. The major application of one such superalloy IN718, a nickel base superalloy, is as material in different parts in gas turbine nearly 35-40% of all components in the temperature between 250°C to 650°C. At such a high temperature sulphur and vanadium as impurities in fuel oil get oxidised in SO2 and V2O5. Further, sulphur oxide reacts with NaCl to form Na2SO4. These salts cause high temperature corrosion and stress corrosion cracking of engine components of marine gas turbine. Therefore, LCF resistance of the material becomes an important consideration in design of turbine. Sanders et al studied LCF behaviour of the alloy IN718 in the temperature range from 204°C to 649°C [3]. Mahobia et al conducted strain controlled LCF tests on the nickel base superalloy IN718 at room temperature as well as 550°C and 650°C [4].

For the study Low cycle fatigue behaviour of IN718, 4 samples 2 coated with NaCl salt and 2 uncoated were prepared. These samples were tested at 550°C at (Δԑ/2): ±0.5% and ±0.7% strain amplitudes at frequency 0.3 Hz. The fractured surfaces of all LCF tested specimen studied by scanning electron microscope to understand high temperature corrosion.
2. EXPERIMENTAL METHODS

The material superalloy IN718 for study was received from M/s MIDHANI (India) as solution annealed at 980°C, holding at this temperature for 1½ hours and then air cooled condition in form of 15 mm diameter rod. The chemical composition of alloy IN718 in wt% was Ni-53.30, Cr- 17.91, Nb+Ta- 5.22, Mo-3.10, Ti-1.02, Al-0.54, Co- < 0.05, Si-0.03, Fe-Balance. The alloy IN718 was obtained in solution treated condition. It was subjected to double-aging heat treatment for 720±5°C-8hrs, furnace cooling @ 55°C/hr to 620°C, holding at 620±5°C for 8hrs, and forced air cooling to room temperature. The optical images in peak aged condition for superalloy IN718 is shown in Figure 1.0 at 50X magnification.

![Figure-1.0 Optical micrographs of alloy IN718 in peak aged condition](image)

For LCF tests 4 cylindrical samples from the heat treated blanks were prepared which have 30 mm length of 12 mm diameter for trenched ends, 14 mm length of 4.5 mm diameter gauge section, 17 mm shoulder radii. Out of these 4 samples 2 samples were coated with 100 wt% NaCl salt of uniform thickness between 2.5 to 5 mg/cm² [5,6]. The salt coated sample was kept in vertical position in furnace for 8 hrs at temperatures 550°C to check the adherence of the salts at this temperature during LCF testing Figure 2.0.

![Figure-2.0 LCF sample in furnace for 8 hrs at temperatures 550°C](image)

Both coated and uncoated samples were tested for low cycle fatigue study using a computerized Servo Hydraulic MTS testing machine Model number 810, 50kN capacity with FlexTest40 digital controller interface equipped with a split electric resistance heating furnace of Model 652.01, Serial number 0114704 with temperature control of ±2°C accuracy. Low cycle fatigue tests on both salt coated and uncoated samples were done in air with fully reversed stress cycle of total strain controlled mode. Tests were performed at temperature 550°C at 0.50%, and 0.70% strain amplitudes with 0.3Hz constant frequency. Strain was controlled by means of a high temperature MTS extensometer (Model 632.53F-14) which was mounted in gauge section of the LCF sample.
After failure of the fatigue samples their fractured surfaces, round and longitudinal samples from
gauge sections were cut to examine under scanning electron microscope (SEM). For the preparation of
SEM samples a length of 3-5 mm was separated from samples along the gauge length with the help of
diamond cutter. After this these samples were cleaned in acetone to remove oil and dust particles.

3. RESULTS AND DISCUSSION
The adherence checking of salt coating is necessary to ensure that the salt coating will not spall
during the LCF testing at high temperature. The sample kept for 8hrs at 550°C in electric resistance
heating furnace and furnace cooled was examined for adherence of the salt coating. Figure 3.0 shows
the image of sample before and after heating at 550°C. It was observed that the coating was still
adhering to the sample.

Figure 3.0 NaCl salt coated LCF samples (a) before and (b) after Keeping at 550°C

The cyclic stress response can be seen from the literature already published in year 2017 [7]. It can
be concluded from there that at higher strain amplitude (±0.7%) there is softening both in coated and
uncoated samples. In NaCl coated sample at strain amplitude ±0.5% there was stabilized cyclic stress
response from the initial cycle to failure, whereas initial stabilization then mild hardening followed by
cyclic softening was observed in case of uncoated samples.

Coffin Manson plot to both salt coated and uncoated situations is shown using log-log scale in the
literature already published in year 2017 [7]. It is important to mention here that sample at low strain
amplitude (±0.5%) was not failed up to 10^5 cycles in uncoated sample. It is clear from the data that
fatigue life was reduced significantly at lower strain amplitude but there was a little difference in the
life at high strain amplitude between uncoated and coated sample.

SEM fractographs of the NaCl coated samples at strain amplitudes of ±0.5% & ±0.7% tested are
shown in Figure 4.0 and 5.0 respectively. Figure 6.0 shows fractographic images of the uncoated
sample tested at ±0.7% strain amplitude. Since the uncoated sample tested at low strain amplitude did
not fail, fractographic analysis could not be carried out on that sample. Characteristic features of crack
initiation, number of crack initiation points and crack propagation may be seen from these figures. At
low strain amplitude the salt coated samples showed evidence of salt particles deposited at the crack
initiation site suggesting effect of salt coating at test temperature on crack initiation. It was also
observed that there was higher inter striations spacing and formation of numerous secondary cracking.
The SEM images at higher strain amplitude of ±0.7% are shown in Figure 5.0 & 6.0. Multiple crack
initiation sites were observed in the salt coated and uncoated samples which were tested at higher
strain amplitude, Δε/2=0.7%.
Figure 4.0 SEM micrographs of NaCl coated LCF sample, tested at (∆ԑ/2): ±0.50% at 550°C showing (a) complete fracture surface and the crack initiation site, (b) salt particle near crack initiation site, (c) stage-II fatigue crack propagation showing fatigue striations and large number of secondary cracking.
Figure 5.0 SEM micrographs of NaCl coated LCF sample, tested at $(\Delta \epsilon)/2$: ± 0.70% at 550°C showing (a) complete fracture surface and the crack initiation site, (b) salt particle near crack initiation site, (c) stage -II fatigue crack propagation showing fatigue striations and large number of secondary cracking.
Figure 6.0 SEM micrographs of the uncoated LCF sample, tested at (∆ԑ/t/2): 0.70% at 550°C showing (a) diffuse salt particle near crack initiation site, (b) stage-II fatigue crack propagation showing fatigue striations and large number of secondary cracking.
Figure 7.0 SEM micrographs showing surfaces of edge of longitudinal sections of the LCF samples, (a & b) NaCl coated, (c) uncoated sample, at 550°C.

The SEM images of longitudinal sections from gauge section are shown in figure 7.0. The gauge surface near to the edge was found to be severely damaged by formation of pits and small cracks propagated to interior and perpendicular to the loading direction may be seen due to the corrosive environment. The distinct damage of the surface may clearly be seen from the magnified image Figure 7.0b. In contrast smooth gauge surface without surface damage may be seen from the Figure 7.0c in the uncoated sample.

4. CONCLUSIONS

It can be concluded from this study that the NaCl coating was intact at temperature of 550°C. Characteristic features of fatigue failure were found both at low and high strain amplitude like crack
initiation, number of crack initiation points and crack propagation. At low strain amplitude the salt coated samples showed evidence of salt particles deposited at the crack initiation sites suggesting effect of salt coating at test temperature on crack initiation. This clearly shows hot corrosion behaviour due the presence of NaCl salt at 550°C. The uncoated sample was not failing even at 10^5 cycles so its fractographic analysis in not given.

ACKNOWLEDGEMENT

I am thankful to my supervisor Dr. G. S. Mahobia and my co-supervisor Prof. Vakil Singh for their continuous support and encouragement during my M. Tech in Metallurgical Engineering Department IIT, (BHU) Varanasi.

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