# Oxidation and Hot Corrosion Behaviour of Ni-based Superalloy Inconel 718 in $Na_2So_4$-75%$V_2O_5$ Environment at Elevated Temperature

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**Abstract**—*Superalloys are the better candidate materials for the high temperature application due to their superior mechanical strength and higher corrosion resistance at service temperature. Nickel-based superalloys are used as components of gas turbines of jet engines. Since these components are exposed to high temperature, their performance is drastically affected by environmental conditions. The present work is based on the study of the oxidation and hot corrosion behaviour of superalloy named as Inconel 718 at a temperature of 900°C. The kinetics for both oxidation and hot corrosion in molten salt ($Na_2SO_4$ + 75% $V_2O_5$) has been studied and the parabolic rate constant was calculated for both processes under cyclic conditions after 50 cycles. The morphologies of the scale, phases, and compositional analysis were characterized by using techniques such as optical microscopy, field emission scanning electron microscopy / energy dispersive analysis, and X-ray diffraction.*

**Keywords**: *Superalloy, Oxidation, Hot corrosion, Oxide scale*

## Introduction

Squalor of components of boilers, gas turbine, metallurgical furnaces, petrochemical installations are mainly due to the high temperature oxidation and hot corrosion. Super alloys find their largest applications in the gas turbine industry due to their good mechanical properties at elevated temperature [1-4]. The super alloy have been developed to enhance their oxidation resistance by utilizing the concept of selective oxidation which are affected by alloy composition, surface condition, gas environment and cracking behaviour of the oxide scale [5]. A number of investigations on the oxidation behaviour and morphology of scale formation on the super alloy are available in the literature [6-12].According to Stott when a clean component is exposed to an oxygen rich gas, small impinges nuclei of all the thermodynamically stable oxides develop on the surface and collapse rapidly to give a complete layer during the initial or transient stage, all the elements in the alloy oxidize and the amount of various oxides in the layer are approximately proportional to the concentration of the elements in the alloy[13]. Residual fuel oil used in energy generation system contains sodium, vanadium, and sulphur as impurities. During combustion sodium and sulphur react with each other and forms $Na_2SO_4$, Vanadium reacts with oxygen to form $V_2O_5$. These impurities react together to form low melting point complex compounds, known as ash, which deposit on the surface of materials and induce accelerated oxidation (hot corrosion). Corrosion occurs when these molten compounds dissolve the protective oxide layers that naturally form on materials during boiler/gas turbine operation due to high temperature oxidation. The inability to either totally prevent the hot corrosion or at least detect it at an early stage has resulted in destruction of engines/ infrastructures [14,15]. Further there is still a lack of basic understanding of how these super alloys behave in severe ($Na_2SO_4$-75%$V_2O_5$) operating environment. therefore the oxidation and hot corrosion behaviour of Ni based super alloy Inconel 718 have been investigated at 900°C under cyclic condition in air with and without $Na_2SO_4$-75%$V_2O_5$ coatings on the super alloy Inconel 718. The cyclic condition has been chosen to test the alloys in the rigorous conditions which are similar to actual service conditions. The kinetics of corrosion of Inconel 718 was investigated by thermogravimetry technique. XRD and Fe-SEM/EDS were used to characterize the corrosion products.

## Experimental Procedure

### Substrate Material

The Ni based superalloys Inconel 718 was procured from M/s Mishra Dhatu Nigam Limited, Hyderabad, India, in the form of rolled sheets. The nominal chemical compositions of the superalloy are given in Table 1.

**Table 1: Nominal Composition of the Inconel 718 Super Slloy**

| | Chemical Composition (wt%) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Midhani Grade | Fe | Ni | Cr | Ti | Al | Mo | Mn | Si | Co | Nb | P | C | S |
| Inconel-718 | 19.8 | Bal | 17.6 | 0.96 | 0.53 | 3.23 | 0.02 | 0.03 | 0.01 | 4.90 | 0.005 | 0.02 | 0.007 |

## Sample Preparation

The specimen of approximate dimension 20mm×15mm×5mm was cut and polished using Emery paper of 220,400 and 600 grit sizes and later on 1/0, 2/0, 3/0, 4/0 grades. Finally specimens were mirror polished with 1 µm alumina powder suspension on a cloth-polishing wheel before the corrosion run.

## Air and Molten Salt Corrosion Test

Cyclic studies were performed for the superalloy substrates in air for 50 cycles and in molten salt environments ($Na_2SO_4$–75% $V_2O_5$). Every cycle consisted of 1 hour of heating at 900°C in a silicon carbide tube furnace followed by 20 min of cooling at room temperature. A coating of uniform thickness with 3–5 mg/cm$^2$ of ($Na_2SO_4$–75% $V_2O_5$) was applied with a camel hair brush on the preheated sample (250°C). The weight change measurements were taken at the end of each cycle using an electronic balance machine with a sensitivity of 1 mg. The spalled scale was also included at the time of measurement of the weight change to determine the total rate of corrosion. The kinetics of corrosion was analysed from the results of weight change measurements. The samples after 50hrs of oxidation and hot corrosion were analysed by XRD for the phase identification and FE-SEM/EDS for surface morphological and compositional analysis.

## Results and Discussion

## Visual Observation

Visual observation of the oxidized samples has been carried after each cycle. During air oxidation the sample dark grey layer was observed on the surface. The oxide layer was very thin and continues. There was no evidence of spallation of the scale upto 25 cycles. After that there was negligible spallation was observed. On the other hand in case of molten salt the spalling of oxide scale started even after 1$^{st}$ cycle. Scale was porous in nature.

## Kinetics of Oxidation and Hot Corrosion

Fig. 1 shows the specific weight change (weight change per unit are) for the air and molten salt ($Na_2SO_4$–75% $V_2O_5$) environment. From the graph it is clear that the weight change during oxidation and hot corrosion follow the parabolic law. The weight change for air oxidation tests is very less as compared to hot corrosion up to 15$^{th}$ cycle. After 15$^{th}$ cycle onwards the weight decreased due to falling of particles/oxide scale outside the boat in case of molten salt environment. The value of parabolic rate constant kp for both (oxidation) = 1.67x $10^{-10}$ gm$^2$cm$^{-4}$sec$^{-1}$ & (hot corrosion) = 1.85x$10^{-10}$gm$^2$cm$^{-4}$sec$^{-1}$ has been calculated by least square method. The graph between no. of cycles and square of weight change has been also shown in Fig. 2. These results are indicating that the Inconel 718 is resistant to the oxidation in air but having comparative poor performance in case of molten salts.

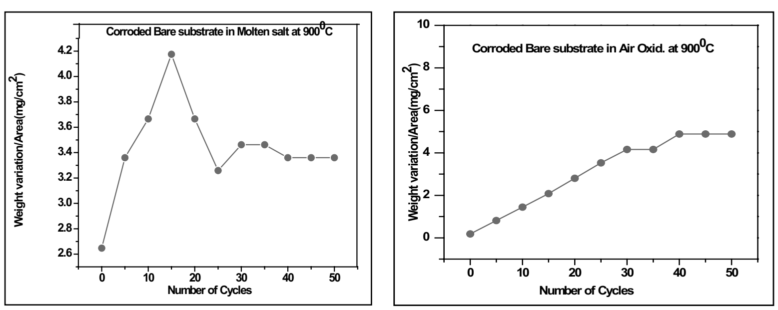



Fig. 1: Graph between Specific Weight Change & No. of Cycles for Oxidation in Air & Molten Salt

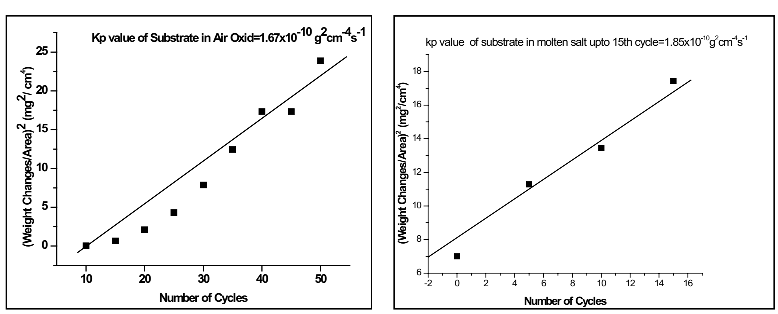



Fig. 2: Kp Values of Substrate in Air Oxidation and in Molten Salt Environment

## X-ray Diffraction Analysis (XRD) of Scale

The XRD graphs for the air oxidation are shown in Fig. 3. The phases identified in case of air oxidation are $Cr_2O_3$, NiO, $NiCr_2O_4$, $Al_2Fe_2O_6$ and some amount of $Fe_2O_3$. Same results have been also reported in literature [14]. The strong peaks corresponding to matrix are showing that the scale is very thin or a little oxidation has occurred even after 50 hours of oxidation.

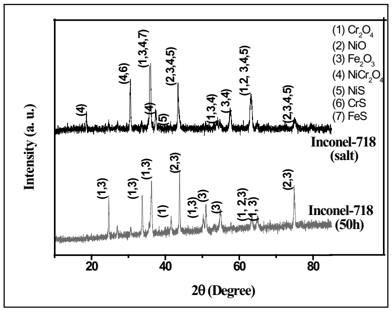
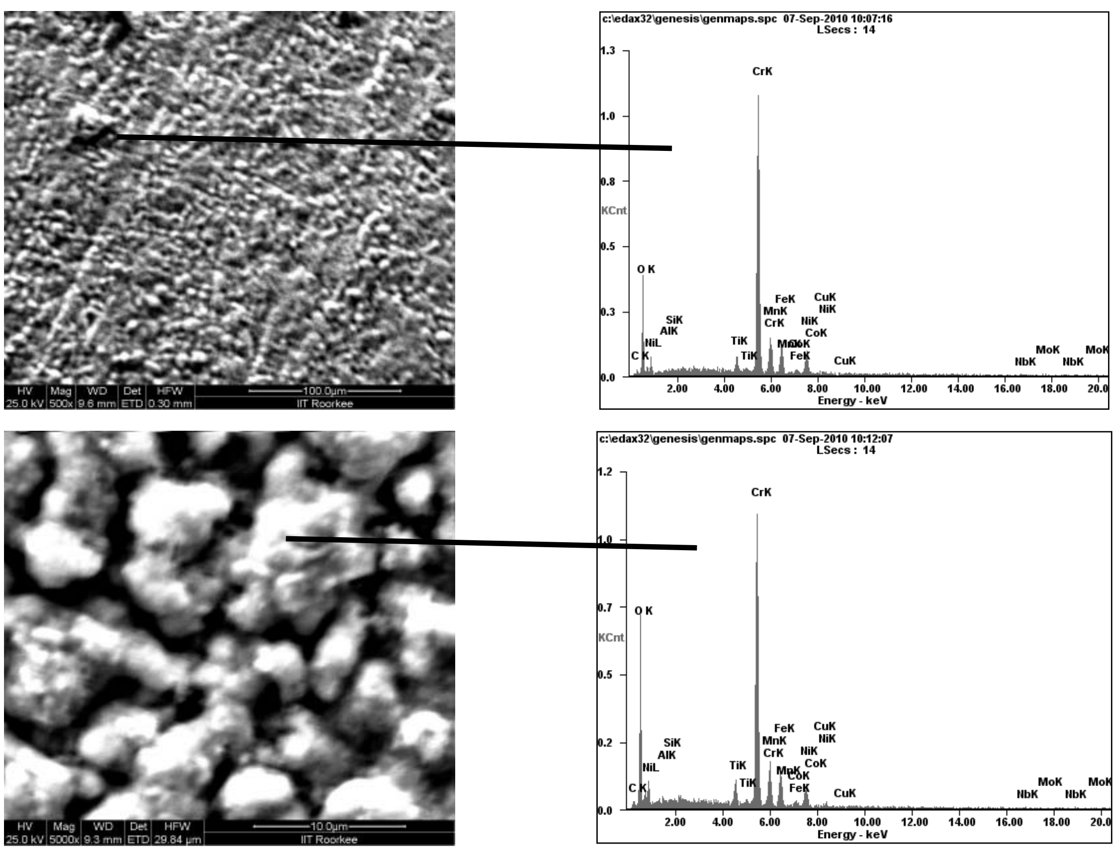



**Fig.3: XRD Pattern of Superni76 after 50hours of Oxidation inAir and in Molten Salt ($Na_2SO_4$–75% $V_2O_5$)**

This result is in accordance with the very small value of parabolic rate constant for air oxidation. The protection against corrosion and oxidation is hence provided by the $Cr_2O_3$ layer. However in case of hot corrosion, the intensity of peaks corresponding to matrix became very less. The major phases identified are: $Cr_2O_3$, and $NiCr_2O_4$.

## Morphology and Composition of the Oxide Scale

Morphology and compositional analysis of the oxide scale in air and in molten salt after 50 hours are shown in Fig. 4(a, b) and 4(c, d) respectively. It is clear from these figures and the XRD that the alloy is chromia former. The main constituent of the scale after 50 hours of air oxidation was the Cr only with minor amount of other elements like Ni, Mn etc. The oxide scale after exposure in air at 900$^0$C for 50 cycles is smooth, continues and dense. In case of hot corrosion the surface as shown in Fig. 4(c, d), is rough with cracked and non-uniform scale. The main constituents of the scale are Cr and Ni with minor amount of other elements like Fe, Mn, Al etc. Same results have been also reported in literature [16].



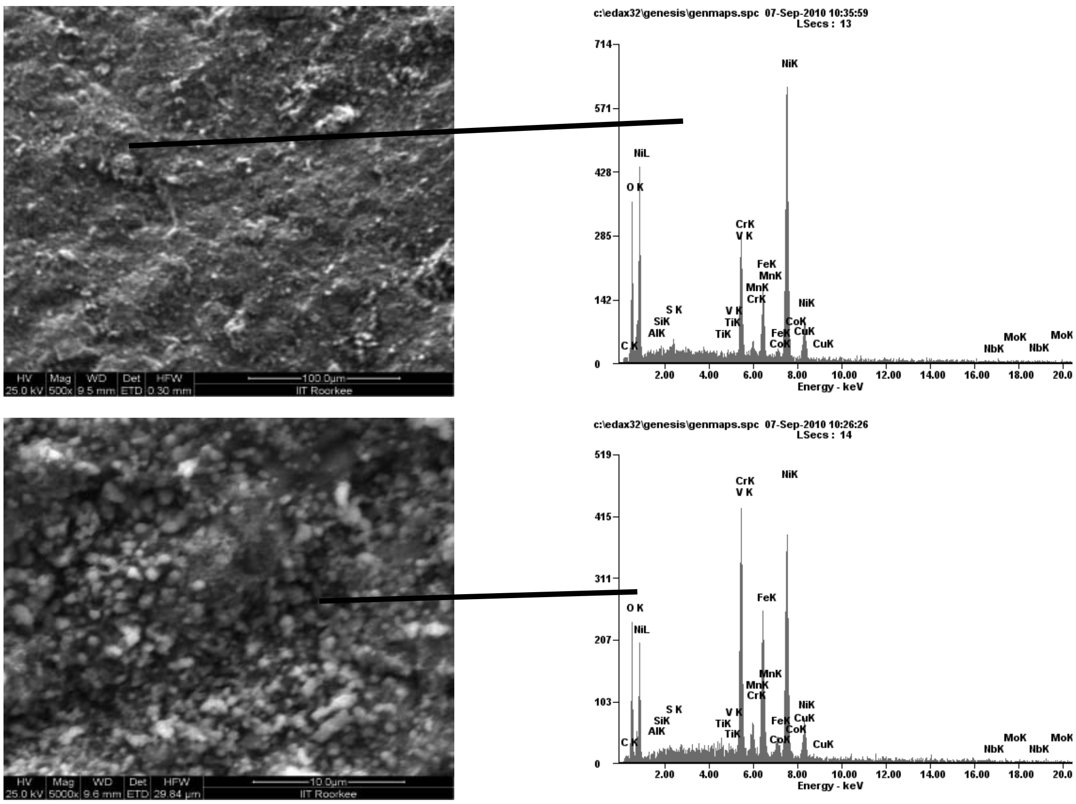



**Fig. 4: Surfece Morphology and Composition Analysis of Oxidized Samples of Inconel 718 at 900°C (a) After 50hr in Air at 500X (b) After 50 hrs in air at 5000X (c) After 50hrs in Molten Salt at 500X (d) After 50hrs in Molten Salt at 5000X**

## Conclusion

Following points can be concluded regarding the oxidation behavior of Inconel 718.

1. Inconel 718 is a chromia former superalloy.
2. The weight gain curve for oxidation in both of the cases follow the parabolic law.
3. The alloy has better oxidation resistance in presence of air as compare to molten salt environment at 900$^0$C.
4. The oxide scale is compact and dense with no sign of spallation for the samples subjected to oxidation in air at 900$^0$C.
5. Ni, Fe, Ti and Al have also migrated from the matrix into the scale formed on the surface.

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