

Corrosion Behaviour of NIMONIC-90 Alloy

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Abstract: Investigations were conducted on hot corrosion of Nickel base, NIMONIC-90 at 675°C and 725°C. The tests were carried out on small test coupons coated with a chemical containing Sodium sulphate and Sodium Vanadate which is representative of the corrosive deposit that usually forms during service in the coal fired boiler and the likes operating in marine atmospheres. Corrosion rates were measured by determining weight loss of the specimen after de-scaling. It was observed that there are three distinct stages in hot corrosion of Nimonic-90 under study. In the first stage the rate of corrosion is rapid and this is mainly due to the formation of corrosion products on new surfaces. In the second stage the corrosive reactions have become stagnant. At high temperatures the corrosive deposit fluxes the protective scale to make it possible. It takes some time before the deposit fluxes and penetrates the oxide scale to attack the substrate. This stagnant stage during which there is no attack can be called “Initiation period”. In the third stage, the corrosion rate accelerates again and the rate of corrosion is increased by over two times that of the initial stage. This stage can be called “Propagation Period”. It is found that propagation stage sets in early at 725°C than at 675°C and also corrosion rate both in the first stage and third stage are high at 725°C as can be expected. Microstructural studies established conclusively that rate of hot corrosion is higher at 725°C than at 675°C.

Key words: Corrosion, Microstructure, NIMONIC alloy

I. INTRODUCTION

Growth of high technology industries, invariably inter linked with availability of special material for aerospace, nuclear power, defence, electronics and other strategic industries have attained a high level of technical perfection which has in turn dictated exceptionally stringent quality requirements on materials. An examination of materials progress since 1942 shows a spectacular series of developments that permitted uninterrupted increase in temperature and operating costs. The developments were both process and alloy oriented, and often a combination of the two. The extremely effective interplay of alloying process with alloy compositions and structures, coupled with excellent supporting scientific studies of structures, properties and stability have given the super alloys an engineering position never dreamed of by their early proponents. The most popular among the super alloys are Nickel-base super alloys, the most widely used for the hottest parts which offer highest strength at elevated temperatures. This property is derived from the strengthening mechanisms like solid solution strengthening, carbide precipitation, inter-metallic precipitation. They are mainly used for high temperature stages of gas turbine blades of combustion chambers, making furnace parts, for turbine discs, high temperature tooling's, and special springs.

Hot corrosion is a form of corrosion that occurs whenever salt or ash deposits accumulate on the surfaces of alloys and alter the environments alloy reactions that would have occurred had the deposit not been present. Deposit modified corrosion or hot corrosion is observed in boilers, incinerators, diesel engines, mufflers of internal combustion processes can vary substantially and significantly affected by the type of the fuel used and the quality of air required for combustion. Hot corrosion is observed to a greater extent in industrial and marine gas turbines compared to aircraft gas turbines. The aim of the present work is to investigate the behaviour of Nimonic 90 super alloy exposed to hot corrosion environments in the laboratory and to determine the extent of hot corrosion at different temperatures and observation of microstructures.

This study is confined to a common grade Nimonic-90 whose composition is given below: Ni-59.0% Cr-19.5% Co -16.5% Al-1.5% Ti-2.5% Mn-0.3% Si -0.3% and traces of C, B, Zr. The alloy chosen for the study has been coated with the deposit of Na₂SO₄ (525 g/lit) and NiVO₃ (87.5 g/lit). This coating is representative of the hot corrosion deposit formed in coal fired boilers. This problem is severe in the temperature range of 650°C-800°C. so the studies have been conducted at the typical temperatures of 675°C, and 725°C.

II. EXPERIMENTAL WORK

The hot corrosion experiments have been performed on test specimens which have been polished to get a rough surface for adherence of the corrosive coatings. Corrosive solution consisting of sodium sulphate and

sodium vanadate was prepared which is a major constituent of hot corrosion in coal fired boilers. The amount and thickness of the deposit have to be maintained consistently and uniformly. A thin uniform coating of the deposit was made on the sample by dipping in the prepared solution for one minute. This process was repeated for four times to get an adherent deposit. Hot corrosion is severe when the service temperature is in the range of 650°C-800°C above 800°C the deposit salts are volatile and hence hot corrosion is not a problem. At low temperature i.e. less than 650°C, significant amount of hot corrosion does not take place because the deposit is not in molten state. The two temperatures selected are 675°C and 725°C. The experiment was conducted for varying times of 40 hrs, 60 hrs, 80 hrs, 100 hrs, 120 hrs and 150 hrs for the above two temperatures. For comparison purpose the experiments were conducted with uncoated samples in a similar manner.

The corroded samples were weighed on "Sartorius" single pan electronic balance at the end of required exposure time in the muffle furnace. The increase in weight was determined and the results were erratic. This might have been due to evaporation losses during the exposure to high temperature. In view of this has been decided that weight loss of the specimen will give a better idea of rate of hot corrosion. After going through literature the procedure for descaling of corroded specimens was arrived at. Descaling procedure is described below.

A. PROCEDURE FOR DESCALING

STEP 1:

A basic solution of KMnO₄ and NaOH is prepared 30gms. of NaOH and 5 gms. KMnO₄ are dissolved in 100 ml of distilled water. The corroded samples are heated in this solution for two hours. The heater voltage is maintained at 130V, so that the heat generated is just sufficient for descaling and boiling does not occur. KMnO₄ present in the solution oxidises the scale by providing the necessary oxygen.

STEP 2:

The acid solution is prepared by mixing 35 ml of HNO₃, 10 ml of H₂O₂ and 55 ml H₂O. The samples are heated in this solution for 45 minutes. The heater voltage was maintained at 130V. Stirring is done so that scale is removed easily. This step is most important in decaling.

STEP 3:

The samples are heated in the boiling distilled water for 15 minutes. This step is to clean the surfaces of the samples. After decaling the samples are weighed on a single pan electronic balance

III. RESULTS AND DISCUSSION

As described earlier the scale formed during the hot corrosion experiments at 675°C and 725°C was dissolved out and weight loss was recorded with time. As can be seen from the Figures 1 and 2 the rate of corrosion increased continuously for the uncoated specimens. Rates of corrosion (Oxidation) have been calculated from the slope of the curve. It is found that rate of corrosion is 0.3 gm/m²/hr at 675°C and that at 725°C is 0.595 g/m²/hr. It is observed from the Figures 1 and 2 that there are three distinct regions in the case of specimens exposed to hot corrosion, both at 675°C and 725°C.

In the first stage the rate of corrosion is high. In the second stage i.e. flat portion of the curve there is no change in weight, which means that there is no attack by the deposit. In the third stage the corrosion rate again increases steeply. In the first stage freshly prepared samples were exposed to high temperature and corrosive atmosphere. Immediately due to the oxidation at high temperature and presence of corrosive coating, the rate of corrosion is high resulting in the formation of chromium oxide scales. The rate is given by the slope which is found to be 0.6g/m²/hr at 675°C and 2.9 g/m²/hr at 725°C. Hot corrosion is a deposit induced corrosion in which the rates of corrosion are aggravated by deposit in the liquid form. These liquid films have to penetrate and flux the protective oxide scales of Cr₂O₃ for further attack to take place i.e. there is some time lag before the actual hot corrosion process is initiated. During this period called "INITIATION PERIOD", the change in weight virtually remains constant. This is characterised by the presence of horizontal portion i.e. stage II in both the Figures.

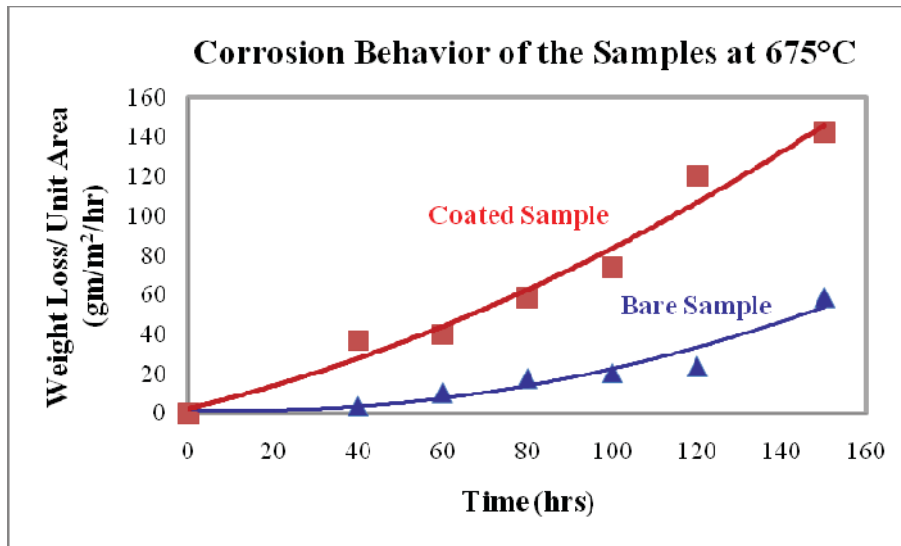


Figure 1: Corrosion behaviour of the samples at 675°C

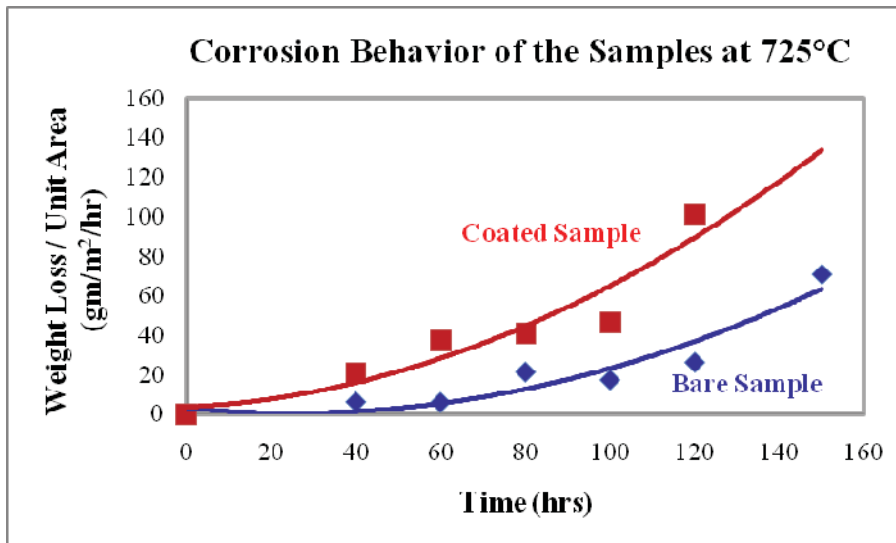


Figure 2: Corrosion behaviour of the samples at 725°C

Once the attack by hot corrosion starts the fluxing of the protective oxide layers takes place and protective oxide becomes porous. As the specimens have been coated with V_2O_5 and Na_2SO_4 which are acidic in nature thus resulting in acid fluxing already described in section 3.3.2. This produces a porous protective oxide. Further corrosive attack increased rapidly as can be seen from the third stage of the Fig. 1 and Fig. 2. This stage can be aptly described as the “PROPAGATION PERIOD”. The rates of corrosion have been calculated and found to be $1.6g/m^2/hr$ and $2.6625g/m^2/hr$ $675^\circ C$ and $725^\circ C$ respectively. It can be also been seen from the figures that the initiation period is shorter at higher temperature followed by the early onset of the propagation period. This is due to higher rates of diffusion, deposit being more fluid and hence fluxing of the protective oxide scale is faster at higher temperatures. The rates of corrosion in the stage III is almost double of that in stage I.

The uncoated samples and the samples exposed to hot corrosion were suitably prepared for metallographic examination. The microstructures are presented in Figures 3 to 8. As can be seen from the microstructure of the raw sample in Fig. 3, a combination of coarse and fine grains is present. The grains are relatively coarser in the sample heated to at $725^\circ C$ for 40 hrs. Some of the grain boundaries are also observed to be attacked by the coating as there is a broadening of the grain boundaries (Fig. 4). It is observed that the attack by the deposit has increased with the progress of time from 40 hrs to 100 hrs. (Fig. 5) and 150 hrs (Fig. 6). Figure 7 shows the microstructure of the sample held at $675^\circ C$ for 150 hrs at a higher magnification. From the above observations

it is clear that the extent of corrosion is more significant at 725°C than at 675°C. The micrograph in the Fig. 8 reveals the scale formation due to hot corrosion on the surface.



Figure 3: Microstructure of bare sample held at 725°C for 100 Hrs at 350X



Figure 4: Microstructure of coated sample held at 725°C for 40 Hrs at 350X

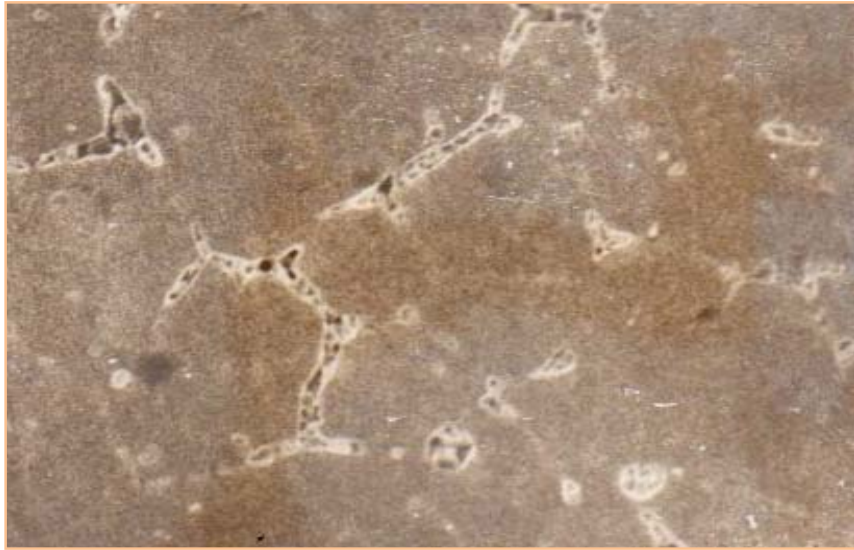


Figure 5: Microstructure of coated sample held at 725°C for 100 Hrs at 350X



Figure 6: Microstructure of coated sample held at 725°C for 150 Hrs at 350X



Figure 7: Microstructure of coated sample held at 675°C for 150 Hrs at 500X



Figure 8: Microstructure showing scale formation on the surface at 1000X

IV. CONCLUSIONS

1. It is established from the laboratory studies that the hot corrosion is characterized by three stages. But the attack by hot corrosion takes place in two steps, first, an initiation stage followed by propagation stage.
2. The attack is more severe at higher temperatures and consequently the period of initiation is reduced at high temperatures and propagation stage starts at an early time.
3. The rate of corrosion has given by the slope of the third stage is 2.66 gm/mm²/hr at 725°C and 1.6gm/mm²/hr at 675°C.

4. Microscopic examination also reveals that temperature enhances the rate of hot corrosion.

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