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Cyclic Oxidation of IN 718 at High Temperatures

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Abstract—*Cyclic oxidation of IN 718 in laboratory air at 750 and 950°C for 20 cycles (1h/cycle) is done. The morphology of the surface and the oxide phases of the scales were characterized by means of weight gain measurements, cyclic oxidation kinetics, scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis techniques. The results showed that as the oxidation temperature is increased, the oxidation rate is increased. Diffusion of substrate elements in the alloy and the inward diffusion of oxygen through the oxide scale control the oxidation rate. Low weight gain and very slow reaction rates of substrate elements at 750°C is observed. At 950°C, a continuous and very thick oxide scale was formed. Significant spallation and volatilization was observed. Formation of* Fe_2O_3 *at 950°C was also an observation.*

Keywords*: Cyclic oxidation, IN 718 superalloy*

Introduction

Adequate experience and knowledge of the high temperature behaviour of the material must be there with Development and Design engineers in order to prevent the various high temperature corrosion problems. Property comparison and ranking of alloy on the basis of performance can be done which thus help in the selection of the material for a particular industrial application [1].

IN 718 is a high-strength, corrosion-resistant nickel chromium alloy and is strengthened by solid solution hardening and precipitation hardening mechanisms [2]. IN 718 has wide range of applications such as: aerospace industries, nuclear reactors, chemical, environmental protection systems, cryogenic applications, and furnaces, where severe temperatures, mechanical stresses, and corrosive environments are encountered. Nb, Ti and Mo are usually added in order to increase the mechanical strength and corrosion resisting properties. Whereas oxidation resistance is provided by a high chromium content wt.% [3–5].

Oxidation is a form of corrosion degradation of the material that usually occurs when the material is exposed to air or oxygen. Sulfur dioxide and carbon dioxide, which have relatively low oxidation potential, can also be the environment for the oxidation. Oxidation can also be defined as the formation of the oxide scale on the surface. If the oxide scale formed is thin, slow growing, and adherent, protection of the substrate from further oxidation is provided and if spallation of the scale occurs frequently, the metal get consumed and ultimately fails. Gravimetric method is one of the most important methods for measuring oxidation kinetics. Principle of the method is to measure the weight change due to oxidation as a function of time. It involves the exposure of a known area of the sample in the furnace at a fixed temperature (called Isothermal Oxidation) followed by cooling and then measuring the weight change at definite intervals of time using a sensitive balance. This forms one thermal cycle. Experiment is interrupted each time the weight change is to be measured. Due to which, changes in the scale behaviour occur.For example, formation of cracks, spallation of scale, etc. However, the advantage is that exposure of many samples can be done at one time and their weight changes can be measured simultaneously. Study and comparison of large number of different alloys with various compositions can be done very quickly in an efficient manner. Main limitation of this method is that thermal stresses arise during cooling and heating at each interruption. The scale formed during the initial period may spall or crack due to thermal stresses. Therefore, an alternative way is to measure the weight change continuously during oxidation which is conducted using a special type of thermo-balances which continuously record the change in weight while the sample is heated [6].

IN 718 is a chromia-forming alloy that develops chromium oxide scale when subjected to high-temperature oxidizing atmospheres in which chromia acts as a slow growing, dense and well-adherent oxide layer and provides protection from the aggressive environment. However, this protective effect gets affected because of cracking and spalling of the scale [7]. Modification of the oxidation behaviour of an alloy can be done through the use of a surface treatment such as polishing, electro-polishing, grinding, shot peening, sandblasting, machining or cold rolling. The effect of surface treatments on oxidation have been studied earlier [8–12].

As a result of surface treatment, energy is stored in the surface region of a metal in the form of dislocations [13, 14]. Therefore, preferential oxidation of the scale-forming elements can result in easier formation of a protective oxide layer for a surface treated metal.

The cyclic oxidation behaviour and scales formed on the IN 718 superalloy at 750 and 950°C up to 20 cycles in laboratory air is investigated in this study by weight gain measurements, oxidation kinetics, scanning electron microscopy (SEM) and X-ray diffraction analysis techniques.

Experimental Procedure

Material

Ni- based superalloy INCONEL 718 was procured from M/S Mishra Dhatu Nigam Limited, Hyderabad, India in annealed and cold rolled sheet form.

Specimen Preparation

Two specimens measuring 25×9×9mm was cut from the rolled sheet of 350×35×9 mm using Power Hacksaw. The samples were polished with SiC polishing paper using successive grades down to 1200, the samples were then rinsed well with distilled water and then cleaned in ethanol and hot air dried (Fig 1). After drying, the samples were stored in polyethylene zip-lock bags.

Cyclic Oxidation Studies

For the cyclic oxidation studies, dimensions and weights of the samples, weight of the crucible, and weight of the sample with the alumina crucible were recorded.

Fig 1: Photograph of the sample before oxidation

Alumina crucibles were prepared at the scheduled test temperature for approximately 2 h until its weight remained constant before the specimen was put into them. The cyclic oxidation tests were performed in a laboratory air at 750 and 950°C up to 20 cycles. Muffle furnace was used for the oxidation tests. A thermal cycle consisted of, a heating period up to desired temperature, followed by a dwell time of 1 h at temperatures 750 and 950°C and cooling for 30 minutes, which was maintained down to room temperature. Twenty identical cycles were applied to the sample (cycles) and the mass change was measured and recorded using electronic balance machine Model CB-120 (Contech, India) with a sensitivity of 1×10^{-3} mg, and then the same samples with their corresponding crucible, were reintroduced into the furnace for the rest of the test. Oxidation rate was calculated as the weight gain per unit surface area.

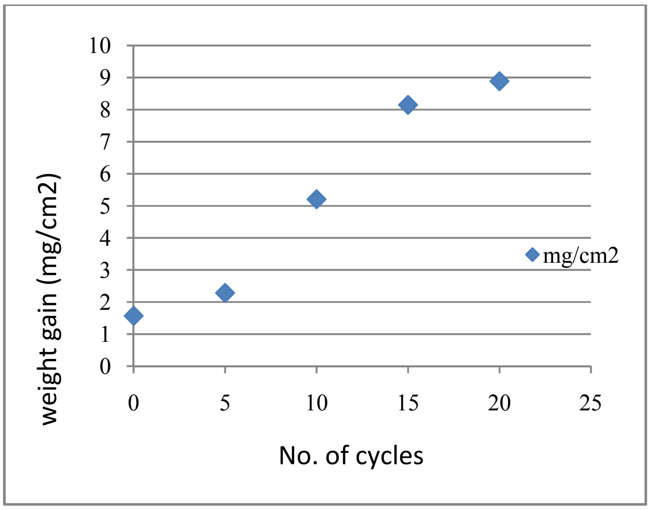
[ΔW/A, in (mg/cm2): where, ΔW: represents the change in the weight, and A: represents the total original area of the specimen], from the measured weight change. The spalled scale was also included in the weight change measurements to determine the total oxidation rate.

After oxidation; the corrosion products on the surface of exposed samples were identified with X-ray diffraction (XRD), using Cuk_{α} (1.5418 A°) in the continuous scan mode in the theta range of $10° \leq 2\Theta \leq 90°$; and the step width was 0.01 with scan speed was 21.6746. The phases present in the patterns were identified using software designed to automatically index the experimentally measured diffraction spectra. For post-exposure-microscopic studies, samples were examined using a surface analysis technique: scanning electron microscopy (SEM) which has been employed to investigate the surface morphology formed on the IN 718 superalloy.

Resultsand Discussions

High temperature oxidation kinetics

The cyclic oxidation kinetics of IN718 superalloy at temperatures 750 and 950°C up to 20 cycles is plotted. Figure 7, shows the linear plots corresponding to the square of weight gain per unit area (Δw in mg/cm^2) as a function of no. of cycles.



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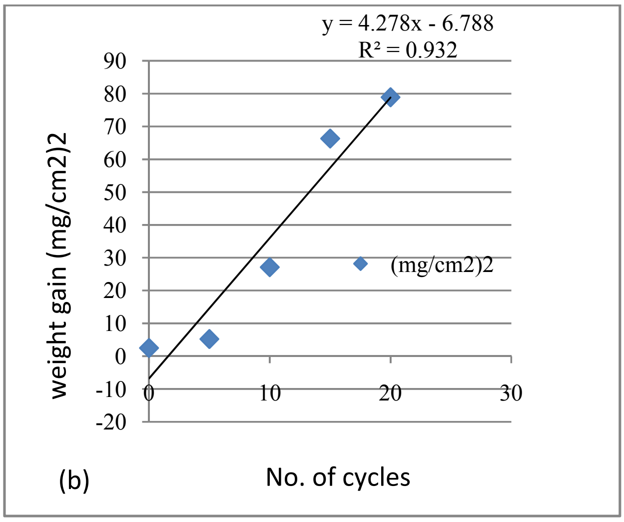

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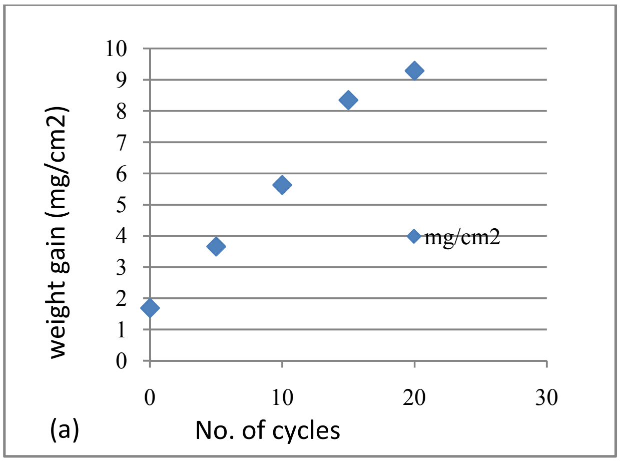

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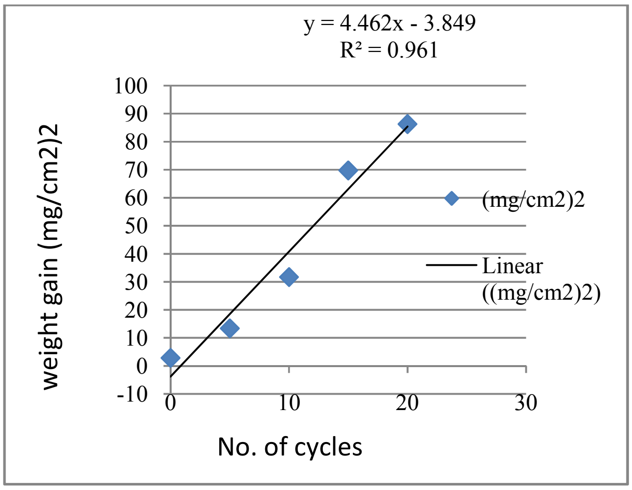


(d)

Fig 2 : (a) Weight changes per unit area (mg/cm^2) versus number of cycles for IN 718 upto 20 hours at 750°C (b)Square of weight gain per unit area ((mg/cm^2)^2) versus no. of cycles for IN 718 upto 20 hours at 750°C (c) Weight changes per unit area (mg/cm^2) versus number of cycles for IN 718 upto 20 hours at 950°C (d)Square of weight gain per unit area ((mg/cm^2)^2) versus no. of cycles for IN 718 upto 20 hours at 950°C

Fig 2 shows the obtained data of squared weight gain in a plot of $(\Delta w/A)^2$ as a function of no. of cycles. The straight lines correspond to the parabolic fit. The parabolic rate constant K_p was calculated by a linear least-square algorithm to a function in the form of $(W/A)^2 = K_p t$, where W/A is the weight gain per unit surface area (mg/cm^2) and 't' indicates the no. of cycles represent the time of exposure. At 750°C, the K_p was 118.8×10^{-11} $g^2cm^{-4}s^{-1}$ but at 950°C, it becomes 123.94×10^{-11} $g^2cm^{-4}s^{-1}$

It can be seen from the Fig 2 that in case of 750°C after 20 cycles, the square of weight gain per unit area is 80g^2/cm^4, while the square of weight gain per unit area is near about 90g^2/cm^4.

At 750°C, as a result of very low weight gains, scatter in data was observed and the oxidation kinetics was very slow so that the experiment must be conducted for longer times in order to detect the change in the weight gains. However, at 950°C, it is clearly seen the weight gain was largely increased. The results show that as the oxidation temperature is increased, the gain in weights is also increased; furthermore, mass gains are higher during the early stage of oxidation, which then become steady. This resulted from the rapid formation and growth of a thin protective oxide film during the early stage of oxidation, which inhibits the diffusion of oxygen and alloy ions in the scale.

Visual Observation

When IN 718 superalloy is oxidized at 750°C (Fig 3), an oxide scale was formed. A black coloured scale is visible which seems to be discontinuous. Most of the surface still appeared shiny gray metal coloured. At 950°C, Fig. 3 shows the surface oxide, which is dark black covering the whole region illustrating that the scale is thicker, extended out of the surface and loosely piled. After 18th cycle, microspalling of the scale was observed. The change in the colour of the scale with increase in the cycles shows the formation of different oxides.

(a)

(b)

(c)

Fig 3: Photograph of specimen along with alumina boat: (a) as received (b) after 750°C (c) after 950°C

SEM Analysis

The SEM micrographs of the specimen before oxidation and surface oxide scales grown by oxidizing upto 20 cycles at temperatures 750 and 950°C are shown in Fig. 4,5, 6.

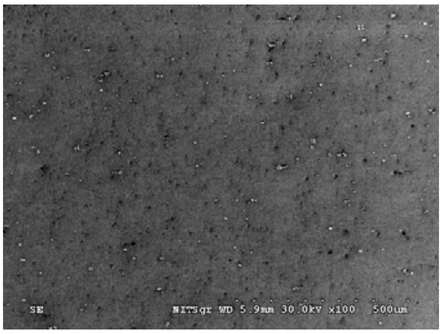
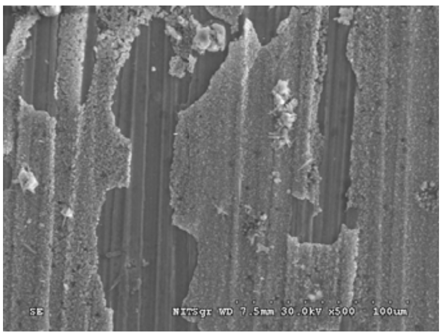
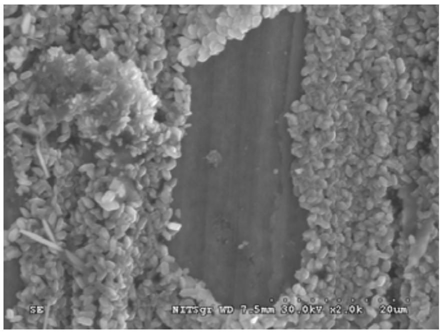


Fig 4: SEM images of the surface of the specimen before oxidation at 100X

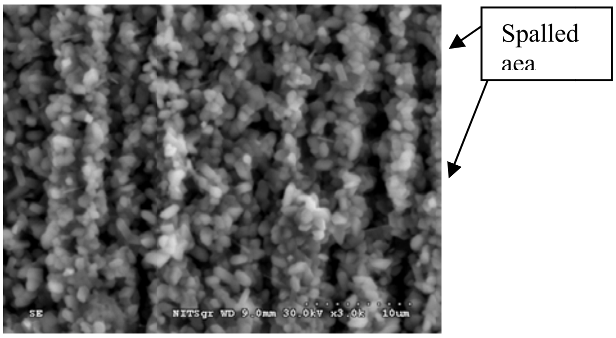


(a)

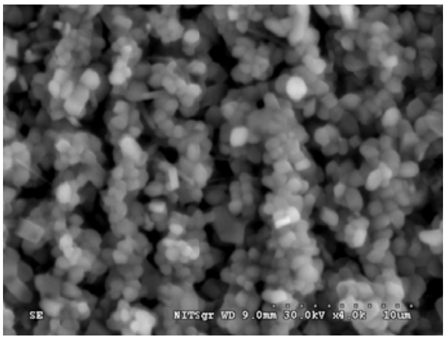


(b)

Fig 5: SEM images of the surface of the specimen after oxidation at 750°C seen at: (a) 500X (b) 2000X



(a)



(b)

Fig 6: SEM images of the surface of the specimen after oxidation at 950°C seen at: (a) 3000X (b) 4000X

At 750°C, fine morphology is observed showing the growth mechanism in 2-D i.e. length and width. But at 950°C, coarse morphology is observed showing the growth mechanism in 3-D i.e in length, width and thickness.

XRD Analysis

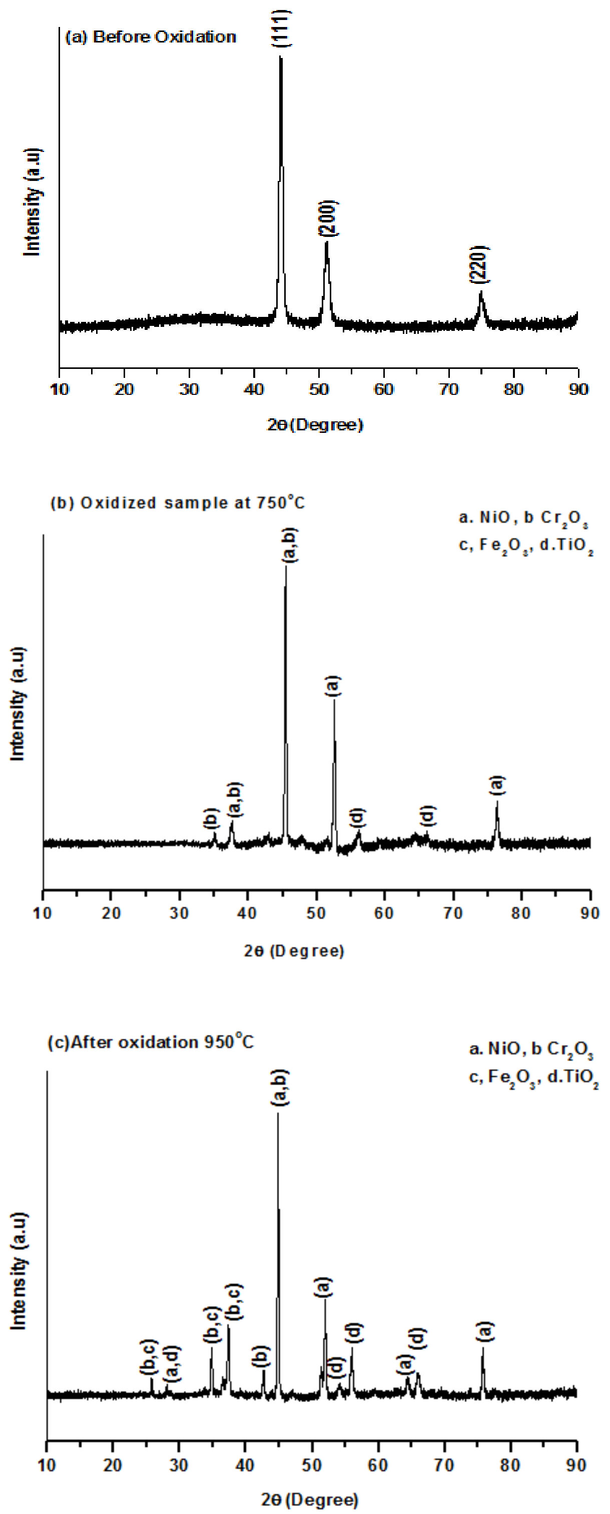


Fig 7: X-ray diffraction plot of the Inconel 718 in (a) as received condition (b) After oxidizing at 750°C (c) After oxidizing at 950°C

XRD (Model: Smart Lab, Company: Rigaku) measurements were made using Cuk$_\alpha$ radiation to characterize the as received as well as after oxidation of INCONEL 718 superalloy. The scan rate was 0.01step/sec and the scan range was 10°to 90°. The XRD results of the as received INCONEL 718 are shown in Figure 7(a). The diffraction pattern has three main reflections on the 2θ scale; 44.16°, 51.03° and 74.82° corresponding to (111), (200) and (220) planes, represents to γ phase (austenite phase). γ phase (austenite phase) corresponds to face-centered cubic Ni-based γ phase of IN-718 alloy. The sharp peak of the XRD reveals the crystalline nature of the alloy. The indexing of the XRD patterns was done as per the reference no. [15].

XRD patterns of oxidized samples are presented in Fig. 7 (b-c). Fig. 7 (b-c) interprets the XRD pattern of the oxidized samples and peaks are indexed with Cr_2O_3, Fe_2O_3, TiO_2 and NiO respectively. Indexing of the diffraction peaks was done as per the reference no. [16].

Conclusion

1. The results show that as the oxidation temperature is increased from 750 to 950°C, the gain in weight due to oxidation is also increased. At 750°C, a black coloured scale is visible which seems to be discontinuous. Most of the surface still appeared shiny gray metal coloured. . However at 950°C, a dark black layer covering the whole region is formed.
2. At 750°C, fine morphology is observed showing the growth mechanism in 2-D i.e. length and width. But at 950°C, coarse morphology is observed showing the growth mechanism in 3-D i.e in length, width and thickness
3. The diffraction pattern represents γ phase (austenite phase) which corresponds to face-centered cubic Ni-based γ phase of IN-718 alloy. The sharp peaks of the XRD reveal the crystalline nature of the alloy.
4. The X-ray diffraction analysis performed on samples oxidized at 750°C revealed that, the oxide phases were found to be mainly NiO with Cr_2O_3 and a small amount of TiO_2 (rutile). Whereas, in case of 950°C, small amount of Fe_2O_3 is also observed in the peaks.

References

1. M. S. Pampana, MSc. Thesis, Louisiana State University and Agricultural and Mechanical College, August 2004.
2. www.specialmetals.com. Accessed 2001.
3. X. S. Xie, J. X. Dong and M. C. Zhang, Materials Science Forum 539–543, 262 (2007).
4. Il. Ho Kim and S. I. Kwun, Materials Science Forum 486–487, 109 (2005).
5. H. Park, H. Kim, Y. Huh, M. Kim, S. Park, J. Koo and C. Seok, Key Engineering Materials 353–358, 523 (2007).

6. A.S. Khanna, Corrosion Science and Engineering, Indian Institute of Technology, Bombay, India, High Temperature Oxidation
7. S. Chevalier, G. Bonnet, K. Przybylski, J. C. Colson and J. P. Larpin, Oxidation of Metals 54, 527 (2000).
8. S. Taniguchi, Y. Shibata and A. Murakami, Oxidation of Metals 41, 103 (1994).
9. H. Kawaura, H. Kawahara, K. Nishino and T. Saito, Materials Science and Engineering A329, 589, (2002).
10. V. B. Trindade, U. Krupp, B. Z. Hangari, S. Yang and H. Christ, Materials Research 8, 371 (2005).
11. V. B. Trindade, U. Krupp, B. Z. Hangari, S. Yang, R. Borin and H. Christ, Materials Research 8, 365, (2005).
12. D. Caplan and M. Cohen, Corrosion Science 6, 321 (1966).
13. C. Ostwald and H. J. Grabke, Corrosion Science 46, 1113 (2004).
14. J. M. Rakowski, G. H. Meier and F. S. Pettit, ScriptaMaterialia35, 1417 (1996).
15. Chinmaya P. Mohanty et al., An intelligent approach to optimize the EDM process parameters using utility concept and QPSO algorithm. Eng. Sci. Tech., Int. J. (2016). http://dx.doi.org/10.1016/j.jestch.2016.07.003
16. Anbarasan et al. Effect of Heat Treatment on the Microstructure and Mechanical Properties of Inconel 718. Materials Today: Proceedings 5 (2018) 7716–7724. Department of Metallurgical and Materials Engineering, National Institute of Technology, Tiruchirappalli-620015, India. School of Mechanical Engineering, VIT University, Vellore-632014, India.