Effect of Nitriding on Surface Characteristics and High Temperature Wear Behaviour of Inconel 718 Superalloy

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***Abstract*— Inconel 718 superalloy was nitrided for 10 h in a fluidized bed reactor containing Al_2O_3 particles as the carrier of the ammonia + nitrogen gas mixture in between 375 and 450°C. The structural characteristics of the nitride layers and the effect of nitriding on high temperature wear properties of Inconel 718 superalloy were investigated. A significant increment in surface hardness (from 495 to 1735 HV) was achieved after nitriding treatment carried out at 400 °C. The applied nitriding treatment led to formation of a nitrogen enriched surface layer exhibiting superior wear resistance at 600 °C when compared to untreated state of the Inconel 718 superalloy.**

***Keywords*— Superalloy, Inconel 718, Fluidized bed, Nitriding, High temperature wear resistance**

I. INTRODUCTION

Superalloys are used in a wide range of applications where reliable chemical and mechanical stability at high operating temperatures is a must. They have good corrosion and oxidation resistance and excellent resistance to creep and rupture at elevated temperatures. Hence, they are commonly used in aircraft jet engines, rocket engines, nuclear-power equipment and petrochemical plants. Among the superalloys, Inconel 718 is the most popular one to be used in fabricating turbine blades, discs and other critical parts of jet engines [1,2]. In spite of their excellent properties, Inconel 718 and other superalloys have insufficient wear resistance at high operating temperatures [3]. Since conventional liquid lubrication becomes ineffective at such high operating temperatures, an alternative engineering solution, in the form of a modified surface, is essential to prevent severe wear [4]. Thus, surface treatments that can improve the tribological behaviour have high potential to increase performance and expand the field of applications of superalloys.

Nitriding, which is an effective surface engineering solution for many ferrous alloys utilised in wear related applications, have also been applied to superalloys by utilisation of plasma processes [3,5-9]. Aw et al. [3], who nitrided Inconel 718 superalloy in between 550 and 750 °C, obtained the maximum hardness (1300 HK) from a CrN enriched surface layer with a depth of 8 µm after nitriding at 600 °C for 9h. In a follow up study [5], they observed that nitriding under these conditions degraded room temperature wear resistance of Inconel 718 superalloy. Leroy et al. [6] stated that nitriding of Inconel 690 at low temperature (<400 °C) leads to the formation of CrN free nitrogen solid solution (expanded austenite, γ_N) layer. Sun [7] studied plasma nitriding of Inconel 600 at temperatures between 400 and 600 °C and reported that maximum depth of the nitrided layer was achieved at 450 °C and a further increase in temperature reduced the layer depth. Also, formation of the CrN precipitates was observed after nitriding at 600 °C. Singh and Meletis [8] studied plasma nitriding of Inconel 718 superalloy at relatively low temperatures and reported that modified surfaces were composed of CrN precipitates and γ_N phase. CrN precipitation was enhanced at the expense of γ_N phase when the treatment temperature increased from 450 to 490 °C. They also stated that nitrided surfaces exhibited significantly higher wear resistance than untreated Inconel 718 superalloy under sliding contact at room temperature and nitriding at low temperature (450 °C) had a beneficial effect on the corrosion resistance. Dahm et al. [9] studied plasma nitriding of an Inconel 601 alloy between 350 and 525 °C for 5 h and reported that nitriding produced surfaces comprising γ_N phase at 350 and 400°C. However, at 450 and 525 ◦C CrN-bearing surface layers were formed.

Since precipitation of CrN during nitriding potentially degrades the corrosion resistance of Cr - Ni alloys, many attempts have been made to modify austenitic stainless steel surfaces by plasma nitriding processes at relatively low temperatures. Owing to CrN free γ_N formation, a significant increment in hardness and wear resistance with a combination of excellent corrosion resistance is obtained [10]. Although formation of γ_N over austenitic stainless steels is difficult by gaseous nitriding processes [11], some promising results have been obtained in a fluidized bed reactor [12-14], which involves rapid and uniform heat transfer from heating medium to the material surface under hydrostatic pressure generated by fine ceramic particles [15]. To the authors' best knowledge, nitriding of superalloys in a fluidized bed reactor has not been reported, yet.

In this study, nitriding of Inconel 718 superalloy in a fluidized bed reactor was investigated. Nitriding treatments were carried out between the temperature range of 375 and 450 °C for 10 h. Nitrided and untreated superalloys were characterised by using X-Ray Diffraction (XRD) analysis, Scanning Electron Microscopy (SEM) examinations and hardness measurements. The effect of exposure to the high temperature atmospheric ambient on the hardness of nitrided surfaces was elaborated. Sliding wear performance of the nitrided alloys was tested at elevated temperature by utilising a pin-on-disc tribometer.

II. Materials and methods

The composition of the Inconel 718 superalloy used in this study was; (wt %): 18.56 % Cr, 17.79 % Fe, 3.02 % Mo, 5.29 % Nb+Ta, 0.96 % Ti, 5.29 % Nb, 0.03 % C, 0.12 % Co, 0.57 % Al and 53.39 % Ni. Disc shaped samples were cut from commercially available bars (10 mm in diameter) which were subjected to either solution heat treatment (AMS 5662) or solution and ageing heat treatment (AMS 5663). Prior to nitriding process, surfaces of the samples were grinded with abrasive papers, polished with 1 µm diamond paste and ultrasonically treated with a chemical which removes the oxide layer from the alloy surface.

Nitriding of the samples were conducted in the fluidized bed reactor chamber containing Al_2O_3 particles as gas carrier and ammonia + nitrogen gas mixture at 375, 400 and 450 °C for 10 h. Initially, nitriding of AMS 5662 samples were carried out in order to determine the appropriate nitriding temperature. Nitriding of AMS 5663 samples were conducted at the predetermined nitriding temperature.

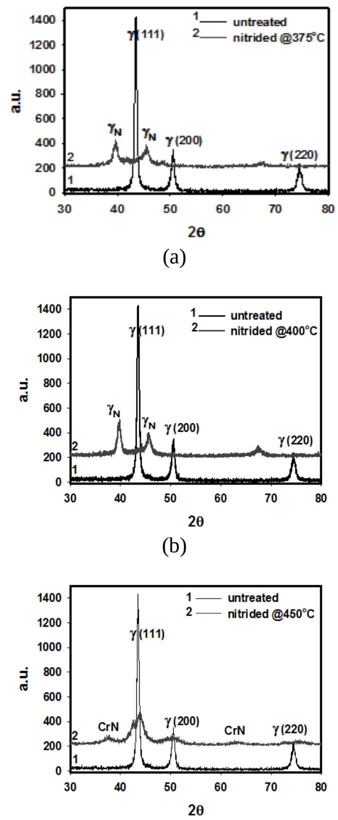
Structural characterisation for phase identification was conducted on the nitrided samples by using glancing incidence XRD technique. A Philips PW 3710 X-ray diffractometer was used for the XRD analysis. Radiation source was CuK_α and the incidence beam angle was 2°. Diffraction angle range was between 30° and 80°, with a step increment of 0.02° and a count time of 1 s. The cross-sections of the nitrided samples were examined with a Hitachi TM1000 tabletop SEM. The nitrided samples were sectioned and prepared by standard metallographic technique. After grinding and polishing metallographic samples were etched in a $HF:H_2O_2:HCI:H_2O$ (1:4:20:25) mixture.

Mechanical properties were determined by hardness measurements and wear tests. Hardness measurements were carried out on the surfaces of the samples with a Vickers pyramid indenter under a load of 250 mN by using a CSM instrumented indentation tester. Wear tests were performed on a CSM pin-on-disk high temperature tribometer at 600 °C. Sliding wear tests were conducted against Al_2O_3 ball (6 mm diameter) under normal load of 2N. The ball slid on a circular path at 2 cm/s linear sliding speed and tests were terminated after reaching 200 m sliding distance. After the wear tests, 2-D profiles of the wear tracks were taken by using a Veeco Dektak 6M profilometer to determine its width and depth. The extent of wear was evaluated according to volumetric wear loss by integrating mean cross-sectional area of the wear track through circular path length. Worn surfaces of the samples were also examined by SEM.

III. Results and discussion

A. Structural characterization of the nitrided surfaces

XRD patterns of the untreated and nitrided AMS 5662 samples are shown in Fig. 1. In the XRD pattern of the untreated alloy, austenite peaks (γ) were observed at different diffraction angles corresponding to different crystallographic planes. Nitriding treatments induced broadening of the γ peaks besides shifting them to the lower angles. The displacement of the diffraction angles was probably caused by the dissolution of nitrogen in γ and expansion of the γ lattice parameters [8]. When nitriding treatments were conducted at 375 and 400 °C, displacement of the γ peaks was more pronounced and this was an implication of the expanded austenite (γ_N) formation during nitriding. After nitriding at 450 °C, CrN peaks were observed on the XRD patterns along with the γ peaks. This indicated that nitriding at temperatures higher than 400 °C induced CrN formation. It has been reported that CrN grows at the expense of the γ_N when Inconel 718 superalloy is processed at temperatures higher than 400 °C [8,9]. Therefore, the samples nitrided at 375 and 400°C were selected for further microstructural characterization studies.



(a)

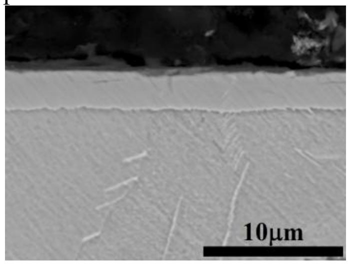
(b)

(c)

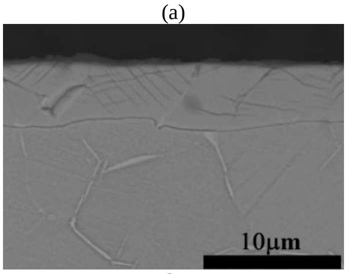
Fig. 1 XRD patterns of the untreated and nitrided AMS 5662 samples. Nitriding temperatures were (a) 375, (b) 400 and (c) 450˚C.

Cross-sectional SEM micrographs of the AMS 5662 samples nitrided at 375 and 400 °C are presented in Fig. 2.

Etching revealed uniform surface layers differing from the underlying substrate. Surface layers appeared to be fully dense and no evidence of local discontinuity has been detected along the interface. The depth of the surface layer was 2-3 µm after nitriding at 375 °C. It increased from 2-3 µm to 5-6 µm when the nitriding temperature increased from 375 to 400 °C.



(a)



(b)

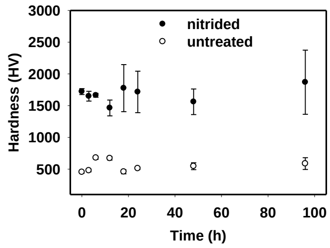
Fig. 2 SEM micrographs of the AMS 5662 sample nitrided at (a) 375 and (b) 400 ˚C.

Hardness of the untreated AMS 5662 sample was 495 ± 31 HV and nitriding at 375°C for 10 h increased surface hardness to 907 ± 38 HV. However, upon nitriding at 400 °C, a significant increment in surface hardness (1735 ± 22) was observed.

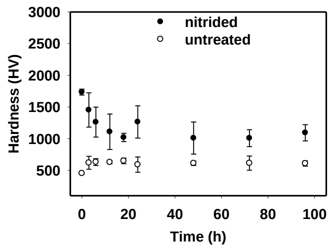
In comparison to the untreated state, higher hardness values obtained after nitriding can be associated with solid solution hardening mechanism. When the position of the most intense (111) γ peak on the XRD pattern (Fig.1) is of concern, the average lattice parameters are calculated as 0.36 nm for the untreated sample and 0.39 nm for the samples nitrided at 375 and 400 °C, respectively. The dissolution of nitrogen atoms resulted in 8 % extension of the lattice parameter of austenite. Thus, alloying of the surface with nitrogen atoms (γ_N formation) provided enhanced surface hardness. In fact, the microstructural observation presented in Fig. 2 indicated that deeper hardened layers generated at 400 °C increased surface hardness significantly. Low surface hardness values measured on the sample nitrided at 375 °C can be attributed to the involvement of softer regions during hardness measurements. Characterisation studies conducted on the nitrided AMS 5662 sample revealed that nitriding treatment carried out in a fluidized bed reactor at 400 °C produced the hardest surface layer due to γ_N formation in the absence of CrN precipitates. Thus, for further investigations the optimum nitriding temperature has been determined as 400 °C.

B. High temperature mechanical stability of nitrided surfaces

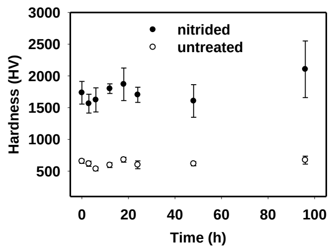
In order to evaluate the effectiveness of nitriding treatment on the characteristics and mechanical stability of the nitrided surfaces, samples cut from AMS 5662 and AMS 5663 quality Inconel 718 superalloys were exposed to atmospheric ambient at elevated temperatures after nitriding at 400 °C. In Fig. 3, surface hardness of the untreated and nitrided AMS 5662 and AMS 5663 samples, which were kept in a furnace at 600 and 700 °C for certain time intervals prolonged up to 96 h is presented. Keeping the nitrided samples (AMS 5662 and AMS 5663) at 600 °C for durations prolonged up to 96 h did not alter the surface hardness significantly. However, hardness varied in between 1500 – 1750 HV, probably, due to relatively high surface roughness. At 700 °C, nitrided surfaces lost their mechanical stability and surface hardness tended to decrease after a short exposure time.



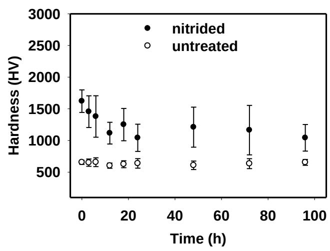
(a) AMS 5662 at 600°C



(b) AMS 5662 at 700°C



(c) AMS 5663 at 600°C



(d) AMS 5663 at 700°C

Fig. 3 Hardness of the untreated and nitrided (at 400 °C for 10 h) AMS 5662 and AMS 5663 samples after exposing to atmospheric ambient at 600 and 700°C for durations prolonged up to 96 h.

In accordance with the general utilisation temperature (less than 700 °C) of Inconel 718 superalloy [16], Fig. 3 confirms that nitriding treatment would provide its beneficial effect on surface hardness at 600°C for extended service durations. Therefore, the effect of nitriding treatment on the wear resistance of Inconel 718 superalloys was investigated at 600 °C for the samples held at 600°C for 96 h. The results of sliding wear tests are shown in Fig. 4 as wear loss volume for the untreated and nitrided AMS 5662 and AMS 5663 samples.

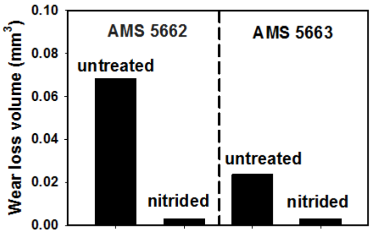


Fig. 4 Wear loss volumes of the AMS 5662 and AMS 5663 samples worn at 600 °C.

In the untreated state, wear resistance of the AMS 5663 sample was better than the AMS 5662 sample due to precipitation hardening effect developed during the ageing process. Regardless of the heat treatment conditions, nitriding treatment carried out at 400 °C for 10 h improved wear resistances of Inconel 718 superalloys significantly. No remarkable difference was observed between the wear loss volumes of the nitrided AMS 5662 and AMS 5663 samples in association with their enhanced surface hardness.

Fig. 5 presents SEM micrographs of the wear tracks. Typical feature of the wear tracks produced on untreated samples (Fig. 5a and 5c) was rough and oxidised appearance indicating progress of wear by sequential growth and removal of the oxide scales. Availability of oxygen in the high temperature (600 °C) ambient accelerated formation of weak oxide scales on the worn surfaces which were easily destructed by shear stresses developed in the next sliding contact event of the counterface [17-19]. On the contrary, the sliding contact of the counterface on the nitrided surfaces seemed to have a polishing effect, without any evidence of weak oxide scale formation on the worn surfaces. Narrow width and smooth topography of the wear tracks developed on the nitrided samples (Fig. 5b and 5d) were clear indicators of limited material removal arising from the high contact resistance of the hard surfaces.

IV. CONCLUSIONS

Nitriding of Inconel 718 superalloy in a fluidized bed reactor at temperatures lower than 450°C produced a hard surface layer consisted of nitrogen solid solution (expanded austenite structure). This surface layer, which preserved its mechanical stability for long durations at 600 °C, provided superior resistance against sliding wear at 600°C.

ACKNOWLEDGMENT

The authors would like to thank to Prof. Mustafa Urgen for XRD analysis.

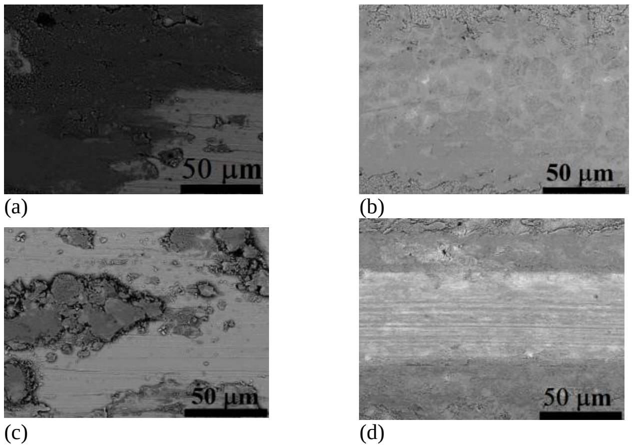


Fig. 5 SEM micrographs of the wear tracks developed during wear tests conducted on Inconel 718 superalloys (a) untreated AMS 5662, (b) nitrided AMS 5662, (c) untreated AMS 5663 and (d) nitrided AMS 5663.

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