Investigations of Surface Dissolution on Fatigue Crack Nucleation in Ni-22Cr-2Fe Alloy

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ABSTRACT: Ni-Cr-Fe alloy specimens were fatigued in a hydrochloric acid solution cell at pH 5.6 \pm 0.1. Results show that the number of surface slip bands forming, extending, and widening increased with the number of cycles. Results also indicated that, initially, cracks initiated as transgranular features but took on intergranular forms as the number of cycles increased.

Keywords: Corrosion, cycle to failure, fatigue, intergranular crack initiation, slip band broadening

I. INTRODUCTION

Fatigue crack initiation in aqueous solutions has been shown to be affected by a number of mechanisms. However, surface pitting [1, 2] is the primary method of attack on many metallic materials that have poor resistance to pitting corrosion of surface oxides, i.e. passive film breakdown [3,4].

The corrosion resistance of the alloy that is the topic of this paper, Ni-22Cr-2Fe, can be significantly affected by cold working, alloy composition, inclusions, heat treatment, precipitates, and, most importantly, the severity and concentration of the test solution and test conditions [5]. Many researchers who have studied corrosion and corrosion fatigue have reported that solution and concentration acidity enhance the pitting corrosion of alloys contains chromium such as stainless steel alloys and nickel based chromium alloy. There are studies reporting the detrimental influence of solutions concentration on the pitting corrosion resistance of alloys contain chromium content such as stainless steels and nickel based chromium alloys [6,7]. Each of these mechanisms and their interaction with fundamental fatigue processes has been recently reviewed with the conclusion that little is actually known about the fundamentals of corrosion and corrosion fatigue in strong solutions [8].

The current study examines the characteristics of corrosion fatigue in an aggressive solution of hydrochloric acid and the evidence of accelerating formation of surface slip bands, pitting, crack initiation, and crack propagation in Ni-22Cr-2Fe alloy specimens.

II. EXPERIMENTAL PROCEDURES

Corrosion test of Ni-22Cr-2Fe alloy in hydrochloric acid solution HCl with pH value 5.6 ± 0.1 was performed prior to fatigue test study as indicated in Figure 1. Axial tension-tension fatigue test were conducted on as received quenched specimens in hydrochloric acid solution as shown in Figure 2. Specimens were carefully polished prior to testing.

III. RESULTS AND DISCUSSIONS

Figure 1 shows polarization test result for Ni-22Cr-2Fe alloy in hydrochloric acid solution HCl at pH 5.6. Test results indicated that corrosion potential (E_{corr}) is -0.4 (V) and corrosion current (I_{corr}) is 5x10³ (NA/Cm²).



Figure 1 Polarization curve of Ni-22Cr-2Fe alloy in diluted HCl solution at pH 5.6.

Corrosion fatigue design is based on use of S-N curves, which were obtained from corrosion fatigue tests in laboratories. The design S-N curve is developed from data points, each point representing a specimen tested up to fracture. It should be pointed out that a few points were deleted because they were anomalous, being well off the trend of other the majority of points. Corrosion fatigue results were summarized in a S-N curve of stress versus number of cycles as shown in Figure 2.

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www.ijmer.com Vol. 3, Issue. 5, Sep - Oct. 2013 pp-2987-2991 For simple fatigue test specimens the testing is performed until the specimens fracture. This means that most of the fatigue life is associated with growth of a small surface crack that grows faster due to hydrochloric acid solution in contact with the specimen surface as the crack size increases until fracture.



Figure 2 Stress versus number of cycles of Ni-22Cr-2Fe alloy fatigued in HCl solution.

An examination of fatigue specimens after testing in the hydrochloric acid solution revealed the generation of a large number of surfaces slip bands with very intense markings in the initial stages of cyclic deformation (Figure 3). This early damage resulted in the formation of slip band cracks oriented nearly normal to the direction of applied stress. Further cycling led to the amplification of many of the surface slip bands. Thus, some surface slip bands remained active in hydrochloric acid solution and these eventually culminated in crack nucleation as shown in Figure 3 and Figure 4.

Intergranular cracking in Ni-Cr-Fe alloys has usually been associated with the application of large number of cycles and consequent increasing corrosion of grain boundaries. These corroded grain boundaries are sites for initiation of intergranular cracking which ultimately leads to fracture. On the other hand, researchers have shown that preferential sites for transgranular initiation, even in long lived fatigue tests [9-11], occur and are attributed to the dislocation substructure associated with the grain boundaries. The intergranular cracking may be associated with alloy ductility and to the large number of cycles. In addition, the intergranular cracking tends to be most dominant in corrosion cracking and corrosion fatigue cracking. The preferential removal of metal atoms associated with dislocations can be expected, as in the case of transgranular attack, in the unlocking of otherwise static dislocation arrays and the subsequent multiplication of dislocation sources. Thus, the state of strain in a small volume of material adjacent to the boundary may be greater than that in the interior [12]. These features are observed to be primarily intergranular along the broadening slip bands as shown, notably in Figure 8.



Figure 3 SEM micrograph showing slip band formation and premature crack nucleation in slips generated from corrosion fatigue of Ni-22Cr-2Fe alloy in HCl solution at 3.29x10⁵ cycles.

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Figure 4 SEM micrograph showing surface slip band broadening on specimen of Ni-22Cr-2Fe alloy in HCl solution at 3.55×10^5 cycles.

As the test progresses, slip bands broaden and further attack is observed (Figure 5) with broadening or development of surface slip bands. Increases in the number of cycles, for example, in Figure 5 and Figure 6, are associated with slip band amplification or an increasing density and broadening of slip bands. At some point, crack initiation and propagation occur.



Figure 5 SEM micrograph showing slip bands and crack nucleation Ni-22Cr-2Fe in HCl solution showing slip intensification at 3.55x10⁵ cycles.



Figure 6 SEM micrograph showing surface slip band in specimen of Ni-22Cr-2Fe in HCl solution, at 3.62x10⁵ cycles.

www.ijmer.com Vol. 3, Issue. 5, Sep - Oct. 2013 pp-2987-2991 ISSN: 2249-6645 Slip band development results in transgranular fatigue crack initiation and growth with a resulting reduction in fatigue life. Slip bands are attacked by the hydrochloric acid solution and primarily intergranular crack initiation and propagation occurs (Figure 7).

It can be seen that broadening or development surface slip bands increases with the number of cycles to failure. For example, Figure 4 and Figure 5 shows slip band amplification or an increasing density and broadening of slip bands with number of cycles to failure respectively.

At some point, crack initiation and propagation show transgranular and intergranular tracks. Slip band development results in intergranular fatigue crack initiation and growth with a resulting reduction in fatigue life. Slip bands are attacked by a diluted hydrochloric acid solution and primarily intergranular crack initiation and propagation occurs mainly along the surface slip bands (Figure 7).



Figure 7 SEM micrograph showing surface slip band in specimen of Ni-22Cr-2Fe in HCl solution, at 3.69x10⁵ cycles.

With increasing fatigue cycles the intergranular cracks and the crack population along slip bands increases. In HCl solution, surface slip bands develop on the specimen surface and are preferentially attacked. This mechanism is responsible for the slip band broadening observed in this study. It is likely that the preferential corrosion fatigue is associated with slip bands. It is also possible that the slip bands are associated with fatigue striations which can be seen especially in Figure 8.



Figure 8 SEM micrograph showing surface slip band in specimen of Ni-22Cr-2Fe in HCl solution, at 3.83x10⁵ cycles.

After fatigue specimen fracture, the fracture specimens were loaded to scanning electron microscopy for surface observations. The area of interest of the current study were mainly two sides of the specimen one represent surface slip bands observation and another side represent fracture surface observation as shown in Figure 9 (a) and (b) respectively.



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Fracture surface of Ni-22Cr-2Fe alloy appeared as ductile fracture like formed by depressions, called dimples, in the microstructure. Intergranular dimple rupture occurs along grain boundaries due to nucleation and coalescence of voids at grain boundaries or at other interfaces such as in slip bands. Final fracture occurs in association with broad slip bands near crack initiation and crack propagation total dimples sites as shown in Figure 10 (a) and (b) respectively.



Figure 10 Fatigue fracture surface of specimen of Ni-22Cr-2Fe in HCl solution, at $3.62x10^5$ cycles. (a) Indicates slip bands and partially fracture surface, (b) ductile fracture surface.

IV. CONCLUSION

Ni-22Cr-2Fe alloy specimens fatigued in diluted hydrochloric acid HCl solution at a pH value of 5.6 developed surface slip bands which broadened and widened with increasing number of cycles. In addition, crack initiation was observed to occur along the slip bands with increasing number of cycles.

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