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Crack Initiation and Crack Propagation of Pre-corroded Ni-16Cr Alloy in 4.5%NaCl Aqueous Solution

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Abstract: - This paper examines the characteristics of corrosion fatigue of a Ni-16Cr alloy workpiece soaked in 4.5%NaCl aqueous solution for a period of one week, followed by corrosion fatigue testing in the same aqueous solution. Corrosion, corrosion fatigue crack initiation mechanisms, crack surface morphology and fractographic analysis are discussed and compared to results from a workpiece fatigued in ambient laboratory air. Experimental results reveal susceptibility to surface pits in the Ni-16Cr alloy workpiece in a 4.5%NaCl aqueous solution, a result that is supported by morphology and fractographic analysis.

Keywords: - *Corrosion, corrosion fatigue, crack initiation, intergranular fracture, pits*

I. INTRODUCTION

Workpieces in service in corrosive environments commonly failed due to pitting corrosion and corrosion fatigue. Typically, such workpieces are made of stainless steels and nickel based alloys with over 12% chromium additions for use in high corrosive environments and high temperature applications. Hence, pitting corrosion and corrosion fatigue have received much attention over the last three decades. It is well reported that the major fracture of workpieces in service is due to a combination of corrosion and fatigue [1-4].

Alloys like Ni-16Cr with Cr content depend on a passive thin film of metal oxide to improve corrosion resistance and are especially susceptible to pitting by local breakdown of the film at isolated sites. Obviously, surface pitting, particularly deep pitting, has a detrimental effect on fatigue life.

Ni-16Cr alloys may not be homogeneous and the passive film may not be homogeneous. In addition commercial alloys contain numerous inclusions and second phases. Thus, even though a passive film is present widely, initiation sites for pitting occur due to compositional heterogeneities. In addition, pitting can occur even in completely homogeneous alloys if other chemical species are present in the environment such as chloride ions [5,6].

Corrosion fatigue failure depends on many variables, including dynamic load frequency, applied stress, and the intensity of the corrosive environment. Researchers discuss which particular variable or variables most influence corrosion fatigue failure at the crack tip [7-9].

Differences in the corrosion fatigue failure surfaces have been observed as between intergranular or grain boundary paths. Recent studies on intergranular grain boundaries have received great attention as a novel process. The results have been extensively used to modify alloy performance and they continue to attract the interest of many engineering materials researchers. Careful design to control the distribution and character of grain boundaries through a thermo mechanical process can eliminate intergranular grain boundary fracture under conditions of corrosion fatigue.

In this paper, characteristics of corrosion fatigue crack initiation and crack propagation processes in Ni-16Cr alloy from test work conducted by the author are summarized and described.

II. EXPERIMENTAL PROCEDURE

Electrochemical tests were performed on a sample of a workpiece of Ni-16Cr alloy soaked for one week in a 4.5%NaCl electrolyte solution.

Before samples were immersed in the solution they were ground with fine sand paper 600 grade and polished with diamond paste. Samples were then immersed and the corrosion potential was monitored and measured. Several readings on each sample were taken. The electrochemical scans were performed to examine the passive film and breakdown film behavior of this alloy prior to corrosion fatigue testing

After soaking for one week in the 4.5%NaCl aqueous solution to create corrosion pits, the workpiece (Figure 1) was placed in a 4.5%NaCl electrolyte in a solution cell for fatigue testing under constant strain amplitude. The corrosion fatigue test results were then compared with fatigue test results performed in ambient laboratory air.

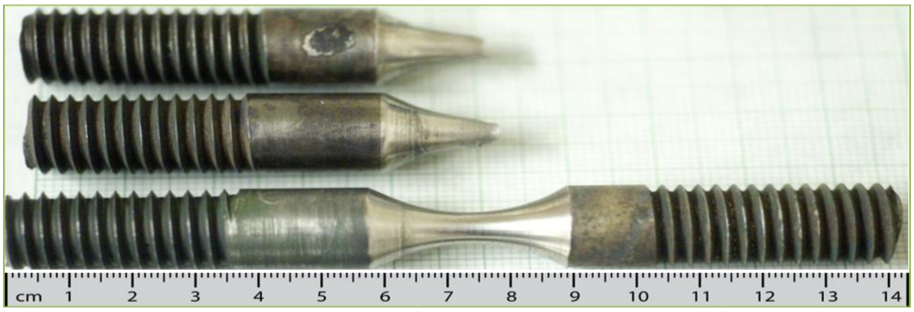

Figure 1 Workpiece tested for corrosion and corrosion fatigue.

III. RESULTS AND DISCUSSIONS

3.1 Corrosion and Corrosion Fatigue Analysis

Cyclic potentiodynamic polarization scans of the Ni-16Cr alloy samples were conducted in a wet corrosive environment of 4.5%NaCl aqueous solution. A typical scan is shown in Figure 2. The workpiece exhibited a passive region with a current density in the range 10^3 NA/cm^2. Following the appearance of this passive region, a small amount of pitting and repassivation was noted as shown by the slight current increase as pitting initiated followed by a decrease in current as repassivation occurs (Figure 2).

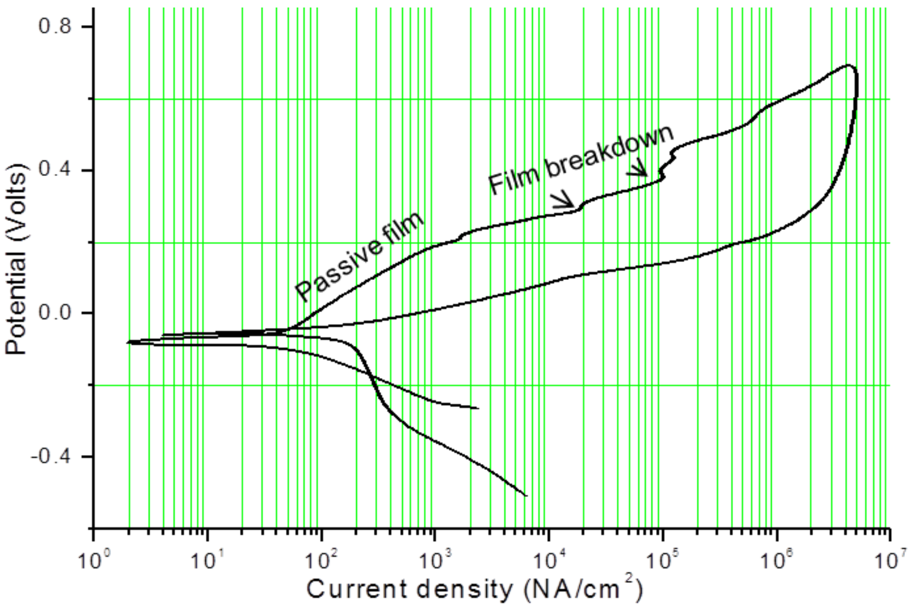

Figure 2 Potentiodynamic polarization test for a workpiece of Ni-16Cr alloy in 4.5%NaCl solution.

The cyclic potentiodynamic data form a large loop curve which indicated that Ni-16Cr is susceptible to pitting corrosion in a 4.5%NaCl solution Figure 3 show S–N diagrams of the workpiece in the aqueous solution at constant strain amplitude. It can be clearly seen from this diagram that the fatigue strength decreases in the solution as compared to a workpiece fatigued in air. The workpiece fractured in aqueous solution at approximately 1.0 x 10^6 cycles, whereas the workpiece in dry air fractured at approximately 4.0 x 10^6 cycles. Thus, the corrosion fatigue strength of Ni-16Cr is reduced by the presence of small amounts of sodium chloride ions and hydroxide ions which promote the formation of surface corrosion pits at which corrosion fatigue is initiated.

Many variables influence the corrosion fatigue strength of Ni-16Cr alloy, including pH, concentration of solution, temperature, applied stress, and surface pits. Surface pits and applied stress are of most concern in corrosion fatigue even though the corrosion fatigue life is increased because of the formation of the protective oxide film [10-11].

Testing to study the influence of surface pits in Ni-16Cr on fatigue strength in corrosive environments is particularly important for the design of workpieces that are to be exposed to such environments and dynamic load when in service. For example, Figure 3 shows S–N diagrams from this study which clearly indicates the marked decrease of fatigue strength in the 4.5%NaCl aqueous solution at the high cycle magnitude up to 1.5x10^6 cycles as compared to the sample tested in ambient laboratory air.

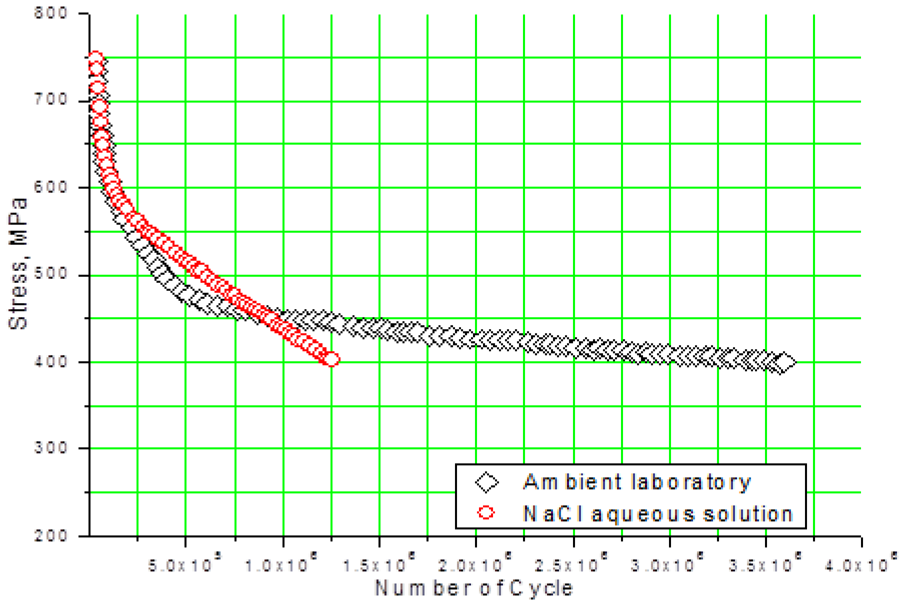

Figure 3 S–N curves of Ni-16Cr alloy in 4.5%NaCl aqueous solution and in ambient laboratory.

Similar work reported on ultrasonic corrosion fatigue tests conducted in NaOH aqueous solution [12] showed that the corrosion fatigue strength of 12%Cr stainless steel decreased with increase of NaOH content. It was also found that the inflection point for 12%Cr stainless steel for various concentrations of NaOH aqueous solution occurs at the higher number of cycles on the S–N curves in 5% and 20% solutions. Moreover, it was clearly observed that the fine corrosion fatigue cracks propagated from the bottom of the blunted corrosion fatigue cracks on the unbroken specimens in 5%NaOH aqueous solution. From these findings it was concluded that the corrosion fatigue strength decreases even in a dilute NaOH aqueous solution if an aggressive environment like 40%NaOH aqueous solution forms at the bottom of fatigue cracks. It is now necessary to clarify the mechanism for the change in the impurity content from bulk solution to the solution in the cracks [12].

3.2 Morphology and Fractographic Analysis

Surface pitting was observed on the unbroken workpiece during electrochemical testing prior to corrosion fatigue. Surface corrosion pits occurred at the grain boundaries in the 4.5%NaCl aqueous solution. The surface of the workpiece was first corroding the grain boundaries and pits were developed at the grain boundaries and within grains as shown in Figure 4. These surface pits were sites for corrosion fatigue crack initiation, followed by crack propagation as shown in Figure 5.

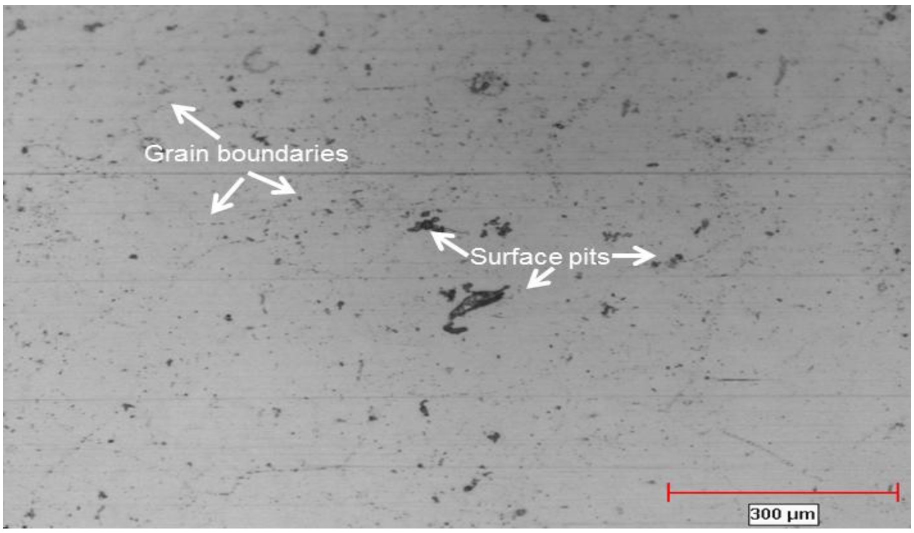

Figure 4 Optical micrograph of surface corrosion pits.

In corrosion fatigue of a workpiece under alternating loading, impurities concentrate at surface roughnesses and in surface pits and can accelerate the corrosion fatigue failure. Generally, it is well recognized that corrosion fatigue fracture of any workpiece occurs under alternating or repeated applied stress and at stress concentrations in the surface pits. However, the stress concentrations mechanism is not yet fully understood. Generally, fatigue fracture of workpieces vibrated under service work conditions can be easily identified on fracture surfaces by beach marks and striations observed by electron microscopy SEM, while corrosion fatigue fracture of workpieces is characterized by corrosion pits from which crack initiation occurs. This initiation pit increase in depth due to stress concentrations accumulated within pits while the pits are in contact with the aqueous solution. Moreover, secondary micro-cracks initiated at corrosion pits are observed at the surface near the primary corrosion fatigue crack initiation sites. Figure 5 shows fine secondary micro-cracks associated with very small corrosion pits.

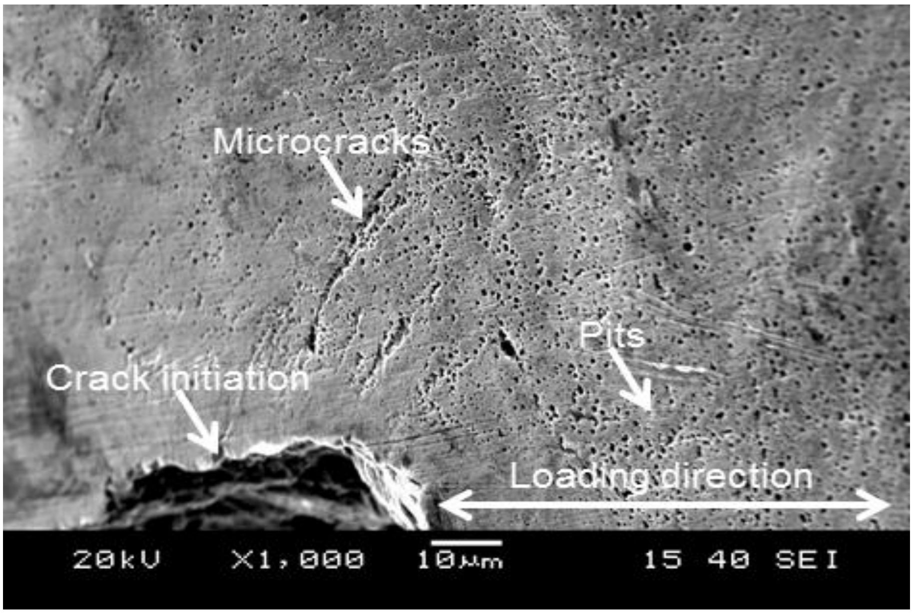

Figure 5 SEM micrograph showing numerous very small surface pits. Fracture initiated at pits.

Crack initiation is observed at grain boundaries Intergranular fracture surfaces are most frequently observed at the corrosion fatigue crack propagation sites. Intergranular fractures at grain boundaries are shown in Figure 6. The formation of intergranular fractures in the Ni-16Cr workpiece was predominantly observed on fracture surfaces.

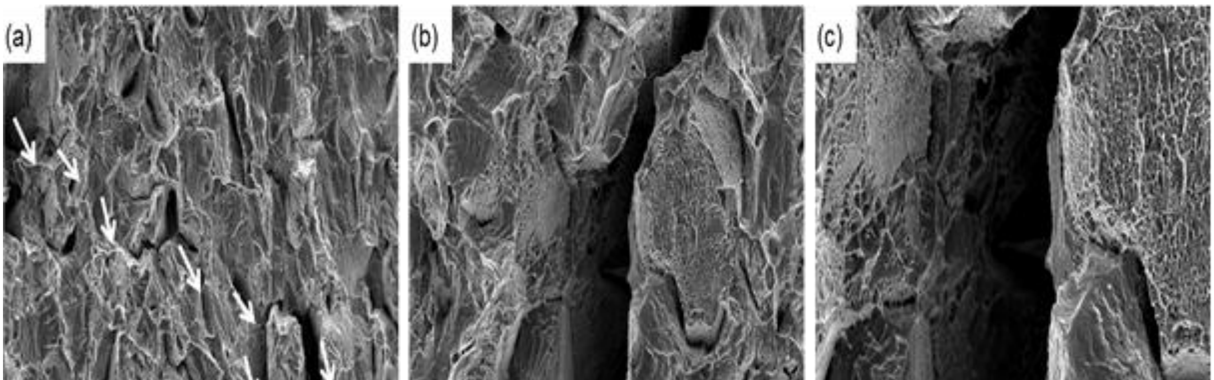

Figure 6 SEM micrographs showing intergranular fractures of Ni-16Cr alloy due to corrosion fatigue in 4.5%NaCl aqueous solution at (a) x200, (b) x500 and (c) x1000. Arrow shows crack propagation direction.

The crack initiation from corrosion pits and the characteristic morphology of crack propagation exhibit a ductile fracture-like appearance at the crack tip. The maximum depth of corrosion pits and their size increase with increasing number of cycles. Thus, it can be concluded that the corrosion fatigue process of Ni-16Cr alloy is controlled by initiation and growth of corrosion pits.

The above observations reveal that cracks are initiated at the bottom of corrosion pits where the stress concentration is largest and is, presumably, a electrochemically active region
The depth and size of corrosion pits initiated on the surface of the workpiece was increased by repeated stress. With increasing number of cycles, more pits were initiated and the depth and size of corrosion pits increased. Thus, the surface oxides protective film is weakened until it breaks.

IV. CONCLUSION

In this paper, the corrosion and corrosion fatigue crack initiation process on workpieces of Ni-16Cr alloy in 4.5%NaCl aqueous solution and the morphology and fractographic analysis were described. Potentiodynamic polarization test scans provided valuable information on the susceptibility of surface pits on the workpiece and were in agreement with the observations from SEM morphology and fractographic analysis..

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