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Intergranular Corrosion Fatigue Fracture Surface Analysis of Nickel Alloy

Aezeden Mohamed

Faculty of Engineering and Applied Science, Memorial University, St. John's, NL, A1B 3X5, CANADA

Abstract

The paper presents a systematic investigation of the effects of a corrosive condition on grain boundary corrosion and other deleterious phenomena in a nickel alloys commonly used in nuclear waste containers, chemical refining plants and steam generator tubes. Grain boundary corrosion is investigated with the aid of microscopy. The results show that a concentrated corrosive condition greatly increases the susceptibility to crack initiation and crack propagation along grain boundaries, i.e. intergranular fracture.

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Keywords: Grain boundary; crack initiation; crack propagation; fracture; nickel alloy.

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1. Introduction

Nickel based chromium alloys have a granular structure of grains and grain boundaries. The morphological characteristics of this structure is a function of the type of heat treatment applied, e.g. air cooled and precipitation hardening. Heat treatments can be agents that weaken the alloy's structure through localized precipitation of the α-phase (iron, chromium, nickel) and resultant corrosion [1-4]. Defects such as voids and impurities can also accelerate the occurrence or localization of corrosion and associated stress concentrations that may lead to ultimate fracture [5-7]. Intergranular grain boundary corrosion also reduces grain size and thereby increases the risk of grain dropout. For example, steam generators are often made of nickel alloys, due to their excellent corrosion resistance and serviceability at high temperatures.

When the alloy under sever corrosive condition, grain boundary microstructural defects are susceptible to attack in a concentrated corrosive environment and to the potential for catastrophic grain boundary cracking. For this, the objective of this paper is to investigate the surface fractography of grain boundaries in a nickel chromium iron alloy subjected to corrosive condition.

* Corresponding author. Tel.: 1-770-7799.
E-mail address: amohamed@mun.ca

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2. Experimental Procedure

The material under investigation was air cooled nickel alloy whose chemical composition is (4.36%C, 21.93%Cr, 2.58 %Fe, 9.53%Mo, 2.39%W, and Ni balance).

Stress controlled fatigue test carried out at frequency of 12Hz was conducted on heat-treated specimens cylindrical fatigue test. The gage length specimens were polished and cleaned prior to testing as described in details in previous work [8]. Fatigue test was conducted in corrosive media of ferric chloride solution were performed by enclosing the specimen within a concentric, “O” ring sealed, acrylic glass.

The fatigued, fractured samples were first investigated by scanning electron microscopy SEM and then sectioned, polished and etched for further investigation by disclosed SEM.

3. Experimental Results

Corrosion and corrosion fatigue of nickel alloy in a wet corrosive ferric chloride media. Corrosion results as shown in Figure 1 (a) the cyclic potentiodynamic polarization curve indicating pitting in the passive film and large cyclic polarization loop that indicate alloy susceptible to pitting in this media. Whereas the corrosion fatigue results as shown in Figure 1 (b) the S-N curve.

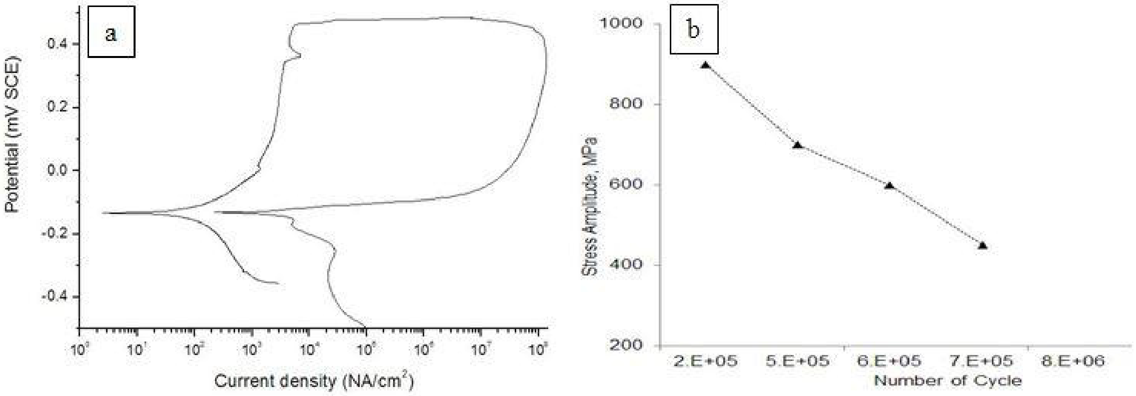

Fig. 1. (a) Cyclic potentiodynamic polarization curve; (b) S–N curve of nickel alloy.

Intergranular or grain boundary fracture is often precipitated (crack initiation) at grain boundary defects. During dynamic fatigue loading the surface microvoids and dimples are found to be sheared and elongated as shown in Figure 2. The crack advance per cycle is rapid. The sample surface is reached through a microscopic growth process of elongation of microvoids and dimples by shear. Some of these voids and dimples may be nucleated by corrosion second-phase particles. These voids and dimples contribute to the shear dimples at high cycles as seen elongated especially those nearer to the final fracture as shown in Figure 2.

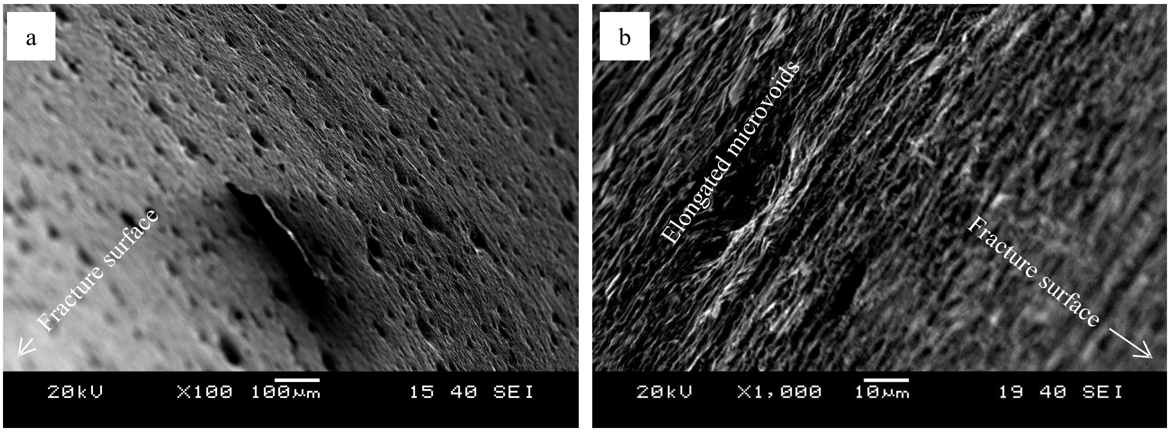

Fig. 2. (a) Surface showing deformed pits during cyclic deformation at 920MPa, Nf = 2.1×10^5 cycles at low magnification; (b) at high magnification.

Figure 3 shows brittle ductile fracture fatigue that was observed at low magnification after a period of cyclic loading. The brittle fracture has a cleavage-like surface fracture resulting from crack initiation. At higher magnification (Figure 3b) the fracture surface shows triangle voids and the striations characteristic of fatigue ductile fracture.

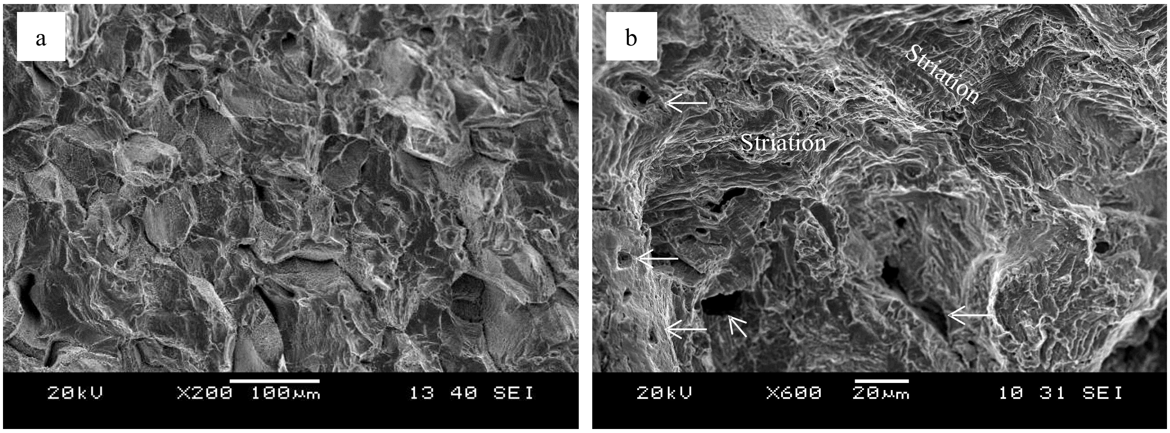

Fig. 3. (a) fracture surface indicates striations and triangular pits deformed at 680MPa, Nf = 5.2×10^5 cycles at low magnification; (b) at high magnification.

Grain boundaries cracking in nickel based chromium alloys are common fracture mode in corrosive conditions and can lead to final fracture.

The brittle grain boundary fracture can be attributed to surface oxides and microvoids that are segregated at grain boundaries. This is often where cracks are initiated and then accelerated to rapid brittle fracture. The slower velocity of ductile fracture is due to high plasticity fracture morphology. Ductile fracture is generated when microvoid coalescence becomes the dominant fracture mode (Figure 4). The transition from the brittle-like cracking to ductile-like cracking is identified by the dashed line in Figure 4.

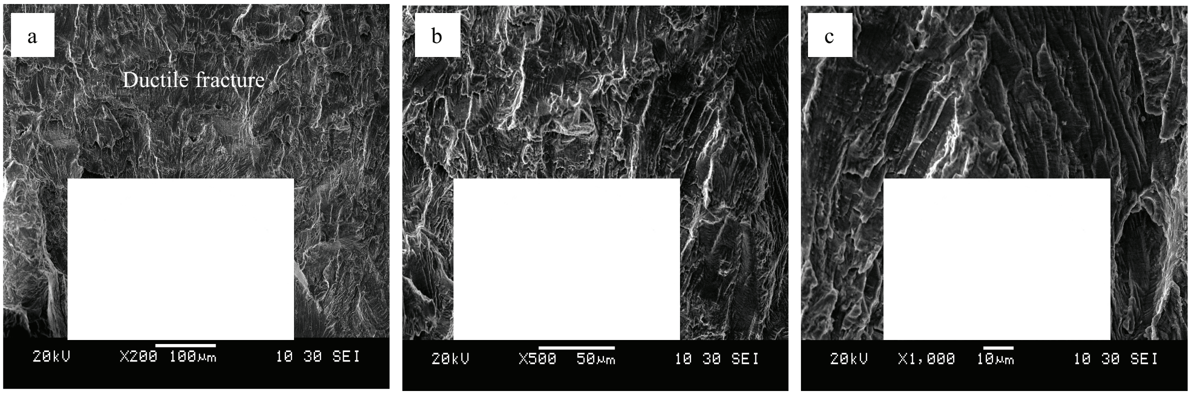

Fig. 4. Fracture surface of a specimen tested at stress amplitude = 600MPa, $N_f = 6.5 \times 10^5$ cycles. Overview of quasi cleavage fracture surface at crack nucleation site and the initial crack propagation. Arrow and transition zone of brittle to ductile fracture.

Crack initiated at sample surface moves towards sample centre as indicated by the arrow and transition zone of brittle to ductile fracture.

Grain boundary cracking is typically associated with intrinsic imperfections in this alloy and with grain boundary weakening where the service condition is corrosive.

This study demonstrates that the factor that increases grain boundary cracking is the presence of precipitates within grain boundaries resulting from air cooling heat treatments that promote segregation of impurities to the grain boundaries. The corrosive condition accelerated final fracture this because due to the direct interaction between sample surface and the corrosive media (i.e., corrosive media must be in contact with the material).

Figure 5 showing initiation sites and direction of fracture propagation zone as in stage I, fine striations zone as can be seen in stage II and coarse striations and intergranular fracture.

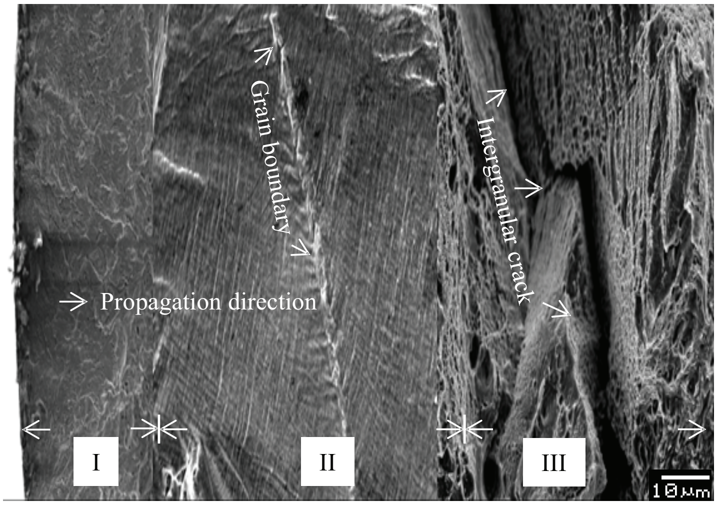

Fig. 5. Fatigue fracture surfaces showing initiation sites, propagation direction, fine and coarse striations as indicated in stages I, II and III respectively

The intergranular fracture is relatively easy to recognize, and easy to initiate because it forms within the grain boundaries, which commonly described as defect of the materials microstructure where is responsible for changing grain orientation and also a source of dislocations when alloy under plastic deformation, this is why has highest state of energy and for this reason when alloy subjected to corrosive media grain boundaries first to be attack due to high state energy prior any grains and subsequently leading to initiation of intergranular cracking.

4. Discussion

The mechanisms of grain boundary fracture in nickel based chromium alloys are also described in the literature [4, 7]. Metallurgical evaluation of a ruptured nickel based chromium alloys in from a normal operating condition determined that the failure mechanism was grain boundary fracture as a result of the corrosive condition of ferric chloride solution. The fracture developed during the cyclical service loading.

Nickel based chromium alloys specimen were subjected to high cycle fatigue in a corrosive condition of SEM fractography of the specimen showed evidence of surface cracking initiated due to the localized stress concentrations as shown in Figure 6 (a). Intergranular crack propagation along grain boundaries was also found Figure 6 (b, c, d). Analysis of precipitates along grain boundaries were products of the corrosive condition. This current study and other literature [7] show that corrosive conditions increase the rate of grain boundary fracture of the alloy. Therefore, the presence of chlorides and oxides on crack surfaces contributes to the development of intergranular fracturing in corrosive condition.

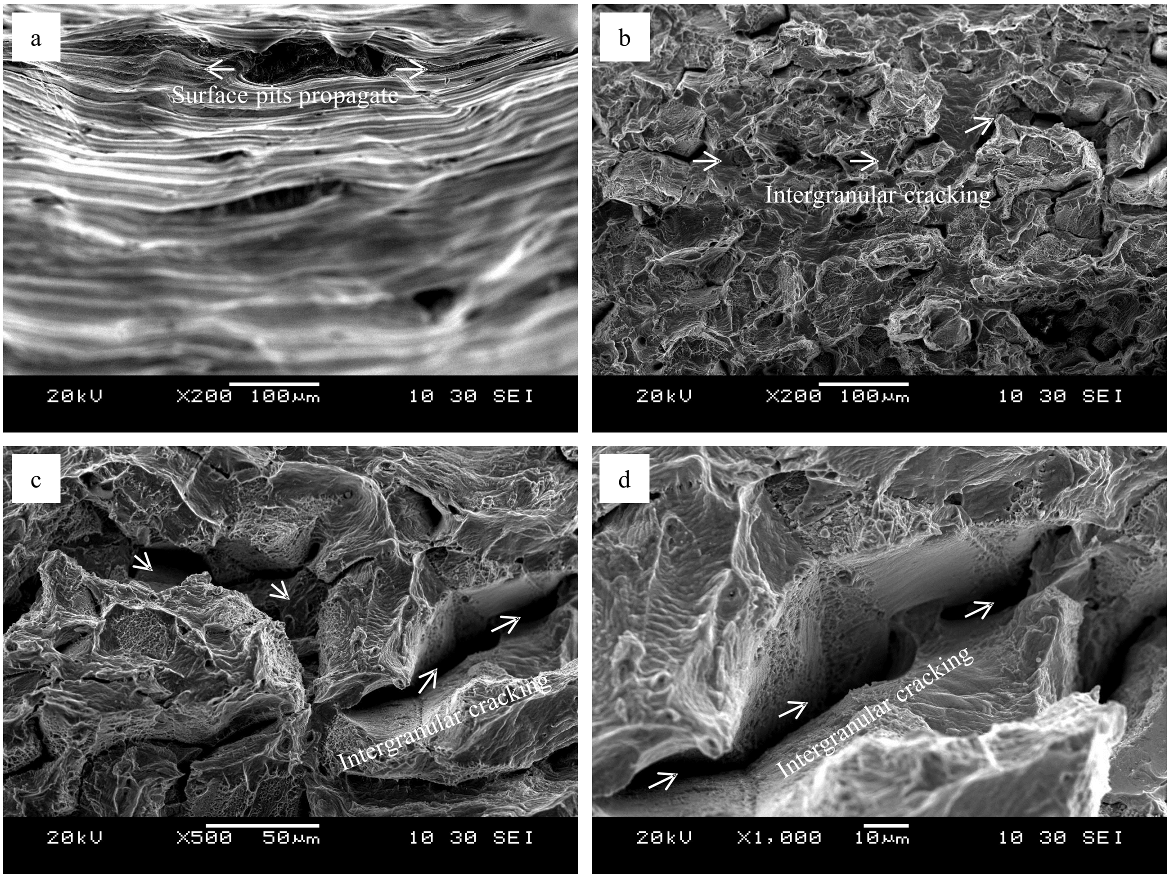

Fig. 6. (a) Surface failure initiation prior to final fracture, (b) fracture propagation along grain boundaries, (c) 500X and (d) 1000X respectively.

Figure 7 reveals the following possible phases: gamma (γ), gamma prime (γ'), M_7C_3, and $M_{23}C_6$ these phases appear white in colour as in Figure 7 and are found to be mostly at the grain boundaries.

From the microstructural characterization it was shown that the corrosive agents primarily attack along the grain boundaries where the carbides and intermetallic precipitates ($\gamma+\gamma'+M_7C_3+M_{23}C_6$) occur. These precipitates determine by the author elsewhere [9]. The morphology of second phase particles along the grain boundary covers a large proportion each boundary (Figure 7). These phases are a product heat treatment with air cooling. The slow cooling rate allows diffusion of intermetallic alloying elements to and along grain boundaries with further growth of the intermetallic precipitates. The degree to which the alloy will be affected by intergranular grain boundary corrosion reaches a maximum with slow cooling rate when the greatest amount of precipitates develops about the grain boundaries (Figure 7).

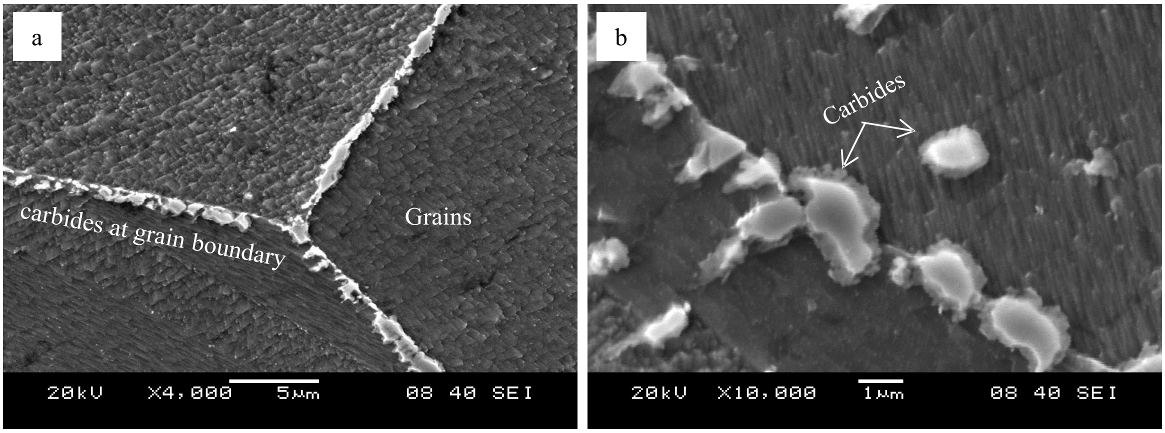

Fig. 7. Intermetallic precipitates develops at the grain boundaries in (a) and (b) at low and high magnification.

5. Conclusions

The current study has established that fracturing of nickel based chromium alloys heat treated by air cooling is by grain boundary cracking and the intensity of its occurrence depends upon the severity of service conditions. The risk of grain boundary corrosion cracking increases with declining cooling rate and reaches a maximum close to the dissolution temperature of the intermetallic precipitates ($\gamma+\gamma'+M_7C_3+M_{23}C_6$), when the greatest amounts of precipitates about the grain boundaries are developed. The localized grain boundaries are attacked by aggressive acidic agents and the extension and enlargement of the widths of the grain boundaries can lead to increasing risk of grain dropout.

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