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Stress corrosion cracking at low loads: Surface slip and crystallographic analysis

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ABSTRACT

Stress corrosion cracking (SCC) of 316L single crystal was studied by scanning electron microscopy and electron backscatter diffraction. SCC could initiate and propagate without surface slip at the normal stress of 20 MPa. The cracks grew on the {1 0 0} planes. Many microvoids appeared at the slip bands and microsteps were present on the fracture surfaces. Consequently, the synergistic effects of microcleavage and local dissolution induced SCC advance on {1 0 0} crystal planes. Microshear was an additional SCC microscopic mechanism at high stress levels.

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1. Introduction

Stress corrosion cracking (SCC) experiments conducted during the last few decades show that environmental fracture in ductile face centered cubic (FCC) materials is the consequence of localized interactions between corrosion and dislocations [1–5]. Namely, local dissolution induces new stress concentrations, favoring the emission of dislocations and the formation of slip steps [3,6]. On the other hand, dislocations move to surfaces or slip steps and disrupt surface protective films. Then dislocations emerge on the surface and freshly exposed metal is chemically attacked [2,7–14]. However, a novel SCC phenomenon has been observed where SCC cracks initiated and grew without any surface slip bands under low loads [15–17]. It is considered that dislocation pile-ups play an important role in the SCC process, and the emergence of slip bands is just an accompanying result at high stress levels, not contributing directly to SCC [15]. As proposed by the corrosion-enhanced plasticity model (CEPM), the increasing localized stresses by dislocation pile-ups and the decreasing critical stress intensity factor, K_{ISCC} due to hydrogen, jointly lead to the initiation of microcracks ahead of the main crack tip [5–6,18–21]. Therefore, it is likely to form an SCC

crack via dislocation pile-ups. Another explanation for SCC without any slip bands is that brittle films formed by anodic dissolution can break at small strains before the base metal yielding [12]. Indeed, the comparison of elastic behavior of the aircraft structural A97075 Al-alloy and bulk Al_2O_3 oxide demonstrates that the fracture strain of aluminum oxide, $\epsilon_f = 0.0007$, is much less than the elastic limit of the aluminum alloy, $\epsilon_{YS} = 0.002$ [12,22]. Consequently, it is also possible that local dissolution takes place at the crack tip and film rupture promotes SCC advance with no slip bands emerging on the base metal.

In general, SCC cracks induced by selective dissolution and dislocation motion often propagate along crystal planes. For FCC metals, the slip planes are {1 1 1}. It is suggested by the CEPM and the slip-dissolution model that SCC sometimes occurs on the {1 1 1} planes of austenitic stainless steel in boiling $MgCl_2$ solutions [5–8,18–21]. Magnin et al. studied SCC of 316 alloys in the 153 °C boiling $MgCl_2$ solution, and suggested that transgranular cracking was related to both microshear on the {1 1 1} planes and microcleavage mainly on the {1 0 0} planes [6]. Li et al. employed etch-pitting and stereographic observations to determine the SCC crystallography of the same SCC system, finding that cracking occurred predominantly on the {1 0 0} planes at low K values [23]. In 304L and 310 stainless steels, SCC cracks occurred primarily on the {1 0 0} planes, while secondary cracks on the {1 1 0} planes were also found [24–26]. Besides, a number of SCC microcracks initiated at the crossing of

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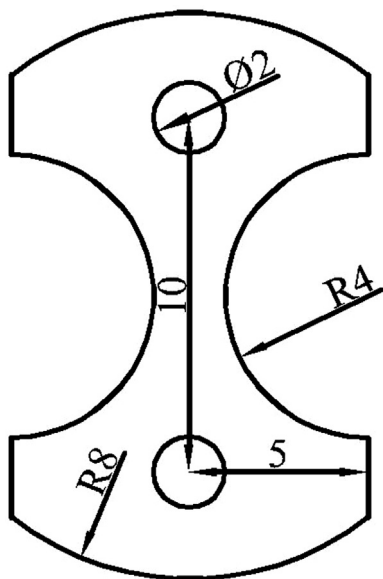


Fig. 1. Schematics of 316L stainless steel single crystal specimens (dimensions are in mm).

two groups of the slip bands and did not follow the $\{111\}$ slip planes [2,15,27–28]. Thus, it is probable for SCC cracks to grow along a few low index planes, except the $\{111\}$ slip planes. In other words, without surface slip, preferential dissolution or microcleavage can occur on some crystal planes, such as $\{100\}$ for 304L and $\{110\}$ for copper due to the lowest surface energy [24,29]. Moreover, in crystals with the small Schmid's factor, the low shear stress well below the critical shear stress value, is applied to the $\{111\}$ slip planes, and no slip takes place [5]. In this circumstance, other crystal planes are possibly subjected to high normal stress and fracture via local dissolution or microcleavage.

Although, lots of research has been conducted on FCC metals slip and crystallography in the past few years, it is essential to correlate SCC and surface slip bands, along with the SCC crystallographic features. In this work, slip bands were investigated on the specimen surfaces with or without short and long SCC cracks. SCC crystallography was characterized by the two-surface trace analysis to determine whether cracks followed the $\{111\}$ slip planes. Finally, microscopic SCC mechanisms of austenitic stainless steel in boiling MgCl_2 solutions are discussed at low and high stress levels.

2. Experimental procedure

316L SS single crystals were used in this study with the following chemical composition: 0.007 wt.% C, 17.00 wt.% Cr, 13.49 wt.% Ni, 2.54 wt.% Mo, 0.66 wt.% Mn, 0.46 wt.% Si, 0.008 wt.% P, 0.0056 wt.% S and Fe balance. The crystals were produced along the $[001]$ direction and machined into 0.7 mm thick specimens with circle holes 2 mm in diameter, as shown schematically in Fig. 1. Prior to experiments, the specimens were annealed at 1050°C for 30 min in argon, water-quenched, ground using 2000 grit emery paper, electrochemically thinned to about 0.5 mm in solution containing H_3PO_4 , H_2SO_4 , CrO_3 , and $\text{C}_2\text{H}_6\text{O}_2$ (glycol), degreased with acetone in an ultrasonic cleaner and washed with deionized water. After the pretreatment, the samples were placed into a glass container filled with a boiling 45 wt.% MgCl_2 solution. Normal stress of either 20 MPa or 40 MPa, calculated in terms of the narrowest section ($2 \times 0.5 \text{ mm}^2$), was applied to the specimens. All experiments were carried out under an open circuit condition using a constant load weight-type apparatus with a cooling system, two 316L SS single crystal pins and two silica grips.

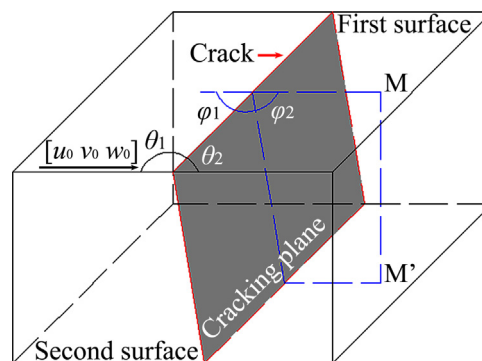


Fig. 2. Schematic diagram of the two-surface trace analysis characterizing the crystallography of the 316L single crystal cracking plane, where M and M' are the labels on the first and second surfaces, respectively. θ_1 and θ_2 are the angles between the crack and the $[u_0 v_0 w_0]$ crystal orientation, while ϕ_1 and ϕ_2 are the angles between the cracking plane and the first surface. The $[u_0 v_0 w_0]$ crystallography of the first surface was determined by electron backscatter diffraction.

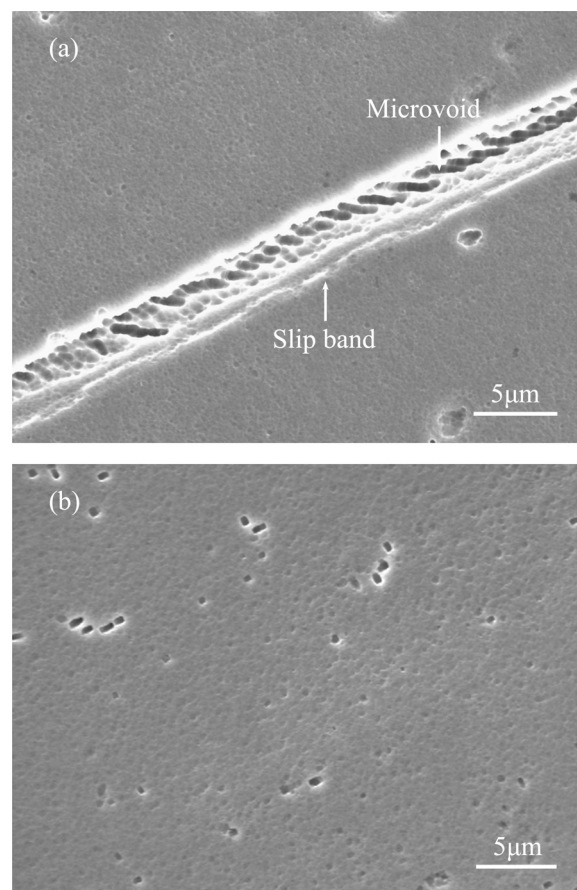


Fig. 3. Surface morphology of the specimens with cleaned off corrosion products, subjected to normal stress of: (a) 40 MPa and (b) 20 MPa. The slip band was corroded and lots of microvoids emerged at the slip band. However, there was no slip band on the surface in Fig. 3(b).

After 200–300 h, SCC cracks grew to a certain size and specimens were taken out from the glass container. Some specimens were ultrasonically cleaned in deionized water as well as 5 wt.% HCl + 2 g/L hexamethylenetetramine mixture to remove corrosion products from the specimen surface. Both cleaned and virgin samples were employed to examine the slip bands and SCC cracks by scanning electron microscopy (SEM). To correlate surface slip and SCC propagation, it was checked whether the slip bands appeared in nucleation areas and near crack tips of short and long cracks. The

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