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Abstract

A chemical cracking test has been used to quickly obtain intergranular stress corrosion cracking (SCC) as it occurs in Alloy 600 wrought metal and EN82 weld metal in deaerated high temperature water environments. The test, referred to hereafter as the doped steam test, involves exposing the specimen surface of interest to 3000 psig (20.7 MPa), 750 °F (400 °C) superheated stagnant steam raised from water that contains 100 ppm each of chloride, fluoride, sulfate, and nitrate sodium salts and to 10 psia (69 KPa) hydrogen partial pressure. Alloy 600 and EN82 bent beam specimens loaded to various known stress levels were exposed to this doped steam environment for periods of one to eight weeks. Threshold behaviors were determined from this test series. For specimens loaded above the threshold stress, SCC occurred in less than one week. Welded specimens with partial penetration EN82 welds were also subjected to the doped steam environment in the built-in crevice associated with partial penetration welds. During this test, cracking occurred in both the weld and wrought materials. The weld cracks initiated at the root and grew through the entire thickness of the weld throat in two weeks. Metallographic sections in the crack region and fractographs of the weld crack surface confirmed the presence of the multiple branched intergranular cracking expected in SCC. The results clearly indicate that the superheated stagnant steam with hydrogen and these four dopants provides a useful environment to assess the tensile stress condition of Alloy 600 wrought metal and EN82 weld metal specimens.

Key terms: chemical cracking test, Alloy 600, EN82 weld metal, stress corrosion cracking

Introduction

A chemical cracking test using doped steam plus hydrogen has been used to quickly obtain intergranular SCC as it occurs in Alloy 600 wrought metal and EN82 weld metal in deaerated high temperature water environments. The test results can be used to compare welding procedures and design features and to locate component regions with tensile stresses greater than the threshold in doped steam. This Alloy 600 and EN82 test serves the same purpose as the stainless steel, boiling magnesium chloride test (ASTM G36-87).

Previous studies[1,2,3] have demonstrated that high pressure (3000 psig (20.7 MPa)) and temperature (750 °F (400 °C)), hydrogen containing (5 to 11 psia (34 to 76 KPa)) steam accelerates intergranular cracking in nickel based alloys. The cracking is further enhanced by adding 30 ppm each of chloride, fluoride, and sulfate (as sodium salts) to the water from which the steam was raised[4]. To further accelerate the cracking, the dopant concentration was increased to 100 ppm and nitrate salt was added. Once this accelerated environment was developed, it was used to assess long term SCC susceptibility of various mockup designs[5,6,7] and to evaluate the effectiveness of several design feature changes[5,8]. A possible mechanism that explains how these steam and hydrogen environments accelerate cracking has been proposed[9]. The dopant concentrations in the water are generally greater than the solubility limits for these dopants in the steam and are a function of steam pressure. At 3000 psig (20.7 MPa), the soluble concentrations of salts were measured to be Cl^- (~90 ppm), F^- (~6 ppm), SO_4^{-2}(~4 ppm), and NO_3^-(~9 ppm) in steam raised from water containing 100 ppm of each dopant. Keeping the salt concentration slightly supersaturated helps maintain a consistent test environment.

The latter test environment of 3000 psig (20.7 MPa), 750°F (400 °C) superheated stagnant steam raised from demineralized water containing 100 ppm each of chloride, fluoride, sulfate, and nitrate sodium salts and with 10 psia (69 KPa) hydrogen partial pressure was used for the testing reported herein and is referred to hereafter as the doped steam test.

Test Description

Threshold Testing

To investigate the threshold stress at which cracks would initiate in Alloy 600 wrought metal and EN82 weld metal in doped steam, sixteen Alloy 600 and sixteen EN82 preloaded, smooth surface beam specimens were loaded in four point bending and exposed to doped steam in an autoclave. The preload stress levels for each material were 0, 10, 20, and 30 ksi (0, 69, 138, and 207 MPa). After each of 1, 2, 3, and 8 weeks, four specimens of each material (one specimen for each stress level) were removed from the autoclave and destructively examined for the presence of SCC.

Welded Specimen Description

Two welded specimens with partial penetration welds, as shown in Figure 1, were also subjected to doped steam. A partial penetration weld is defined as one that has a built-in crevice as a result of the weld geometry. Both welded specimens were fabricated using the same materials and the same machining and welding procedures. Wrought Alloy 600 material was joined with an EN82 attachment weld using tungsten inert gas. As a result of weld geometry, root extensions occurred at the location shown in Figure 1. Stresses in the welded specimens during the doped steam test were welding-induced residual stresses and pressure stresses from the 3,000 psig (20.7 MPa) steam pressure. In this application the pressure stresses were small (approximately 2,000 psi (13.8 MPa)) whereas the welding-induced residual stresses in this highly restrained weld geometry were believed to be approximately equal to the room temperature yield stress of the weld metal. Only the built-in crevice was subjected to the doped steam environment.

Test Apparatus

Figure 2 is a schematic of the test set up. Since the welded specimens were too large to fit into available autoclaves, the heat to maintain the 750 °F (400 °C) steam temperature was provided by an oven which contained the welded specimens, silver palladium cells, and doped water inventory. The built-in crevice replaced the autoclave as the pressure boundary. Two silver palladium cells were used to monitor and control the hydrogen content in the steam. The cells, which display a high permeation rate for hydrogen at temperatures greater than 300 °F (150 °C), allowed hydrogen partial pressure to be measured with one cell and hydrogen to be backfilled with the other.

Test Execution

The test start-up sequence was to measure the pH and conductivity of the doped water, perform pretest chemical analysis, leak test the pressure boundary, evacuate the pressure boundary, add doped water, and slowly increase the oven temperature (less than 150 °F/hr. (65 °C/hr.)). The quantity of doped water added was approximately ten percent more than the amount necessary to create the desired steam pressure in the test volume. During the test the temperature was maintained at 750 ± 12 °F (400 ± 7 °C), the pressure was maintained at 3,000 ± 75 psig (20.7 ± 0.52 MPa), and the hydrogen was targeted for 11 ± 1 psia (76 ± 0.0.007 KPa). It was not always possible to maintain the hydrogen partial pressure within these tolerances due to diffusion of hydrogen through the specimen walls. Measured hydrogen partial pressure varied from 3 to 12 psia (21 to 83 KPa). For subsequent tests the hydrogen partial pressure was maintained within desirable limits using a servo controlled system. At preset intervals or at times when the test was interrupted due to loss of pressure, ultrasonic (UT) inspections were performed to determine crack initiation time and size. The UT inspection times are listed in Table 1. Conductivity, pH, and chemistry determinations were made each time the test was shut down. Typical start up and shut down values for pH were 6.5 and 8.0, for conductivity were 1100 µS/cm and 500 µS/cm, and for F, Cl, NO_3, SO_4, and PO_4 sodium salts were 96, 101, 97, 95, and <0.1 ppm and 57, 40, 52, 53, and <0.1 ppm.

Test Results

Threshold Testing

The EN82 weld metal specimens stressed to 8 ksi (55 MPa) did not experience SCC for the eight week test duration. Therefore, the threshold for eight weeks is greater than 8 ksi (55 MPa). The EN82 weld metal specimens experienced SCC after one week of exposure to 15 ksi (103 MPa). Consequently the threshold is less than 15 ksi (103 MPa).

The Alloy 600 wrought metal specimens with no applied stress experienced some shallow surface cracking (0.0015 inches (0.038 mm)) that was concluded to result from a surface condition not driven by the applied stress. Consequently, the zero stress specimens were considered uncracked for the purposes of determining a threshold. At a stress level of 10 ksi (69 MPa), the Alloy 600 specimens did not crack in one week but did crack for all time periods greater than one week. Consequently, for exposure times of two weeks and longer, the threshold for Alloy 600 wrought metal is less than 10 ksi (69 MPa).

Welded Specimen Crack Description

Through weld, multiple branched cracking that followed the large columnar grain boundaries occurred in both welded specimens. Cracking initiated early, as cracks were detected at each inspection after the start of doped testing. Intercolumnar SCC grew through the throat of the weld in 366 hours for Welded Specimen 1 and 252 hours for Welded Specimen 2. The presence of through weld cracks was confirmed by the inability of the built-in crevice to hold pressure and by bubble formation on the outside surface of the weld. Table 1 and Figure 3 show the crack depth for Welded Specimens 1 and 2. The weld length direction is perpendicular to the cross section of Figure 1. The variation in crack depth in the length direction resulted from a nonuniform distribution of residual stress along the weld length.

Metallographic sections showed the following: (1) intercolumnar cracking in the direction of the weld throat, (2) significant crack depth through weld throat and wrought metal, (3) significant variation in crack shape along the length of the weld, (4) multiple branched cracking, and (4) weld root extensions that occur when the weld metal is deposited. The crack morphology determined from

TABLE 1. INTERCOLUMNAR SCC HISTORY FROM ULTRASONIC TESTING		
WELDED SPECIMEN	CUMULATIVE TEST DURATION (HOURS)	MAXIMUM CRACK DEPTH (PERCENT OF CORRESPONDING WELD THROAT THICKNESS)
1	209	71 %
	347	86
	366	90
2	209	69
	252	96

metallographic sections agreed with that of the UT inspections defined above. The metallographic sections as in Figure 4 showed cracks in the weld metal that were ninety percent through the weld throat. Most of the locations investigated had intergranular SCC in both weld and wrought metal as in Figure 4. A few sections had cracking in the wrought metal only. The significant variation in crack path along the length of the weld is demonstrated by Figures 5a and b which show different crack path for two sections spaced only 0.050 inches (1.27 mm) apart. In the wrought Alloy 600, the intergranular SCC was thirty to fifty percent through the wall. The cracks in the weld metal almost always initiated at the root extension as in Figure 5a. The cracks in the wrought metal usually initiated at the root extension but sometimes originated at the smooth surfaces of the Alloy 600 wrought metal.

After evaluating the metallographic sections, the weld pieces were broken out of the mounts, pulled apart to expose the crack surface, and evaluated using scanning electron microscopy (SEM). The fractograph of Figure 6 shows long columnar grain boundaries exposed by intercolumnar SCC and ductile tearing regions caused by pulling apart the weld piece. The ductile tearing occurred at the ligament near the surface of the weld that was not cracked by SCC and at small islands of weld metal surrounded by intercolumnar SCC. These latter features result from the discontinuous cracking characteristic of intercolumnar SCC. These two types of ductile tearing regions are labeled in Figure 6. Near the weld root where intercolumnar SCC initiates, higher magnification fractographs show surface deposits that completely cover the grain boundaries. Near the outside surface of the weld, the surface deposits do not cover the entire surface and are very sparse, since the time of exposure of these grain boundaries is shorter.

Interpretation of Results

The crack patterns in Welded Specimens 1 and 2 define locations of tensile residual stresses greater than the threshold stress for cracking in doped steam. The maximum tensile stresses in the partial penetration weld basically follow a forty-five degree plane starting at the weld root. The tensile stresses in the partial penetration weld are large enough through the full throat thickness to cause cracking through the full partial penetration weld throat. The tensile stresses in the wrought Alloy 600 are large enough to cause cracking though thirty to fifty percent of thickness of this material within the test duration. In locations where there was no intergranular SCC during the doped steam test, it can be concluded that either the combination of pressure and welding-induced residual stresses were compressive or small tensile (i.e., less than threshold stress levels) or that the stresses were relieved as the intergranular SCC progresses in the cracked regions. Intergranular SCC from doped steam tests is very similar to that observed in Alloy 600 studies[10].

Conclusions

A chemical cracking test using doped steam plus hydrogen has been used to quickly obtain intergranular stress corrosion cracking as it occurs in Alloy 600 wrought metal and EN82 weld metal in deaerated high temperature water environments. The test results can be used to compare welding procedures and design features and to locate specimen regions with tensile stresses greater than the threshold in doped steam.

Acknowledgments

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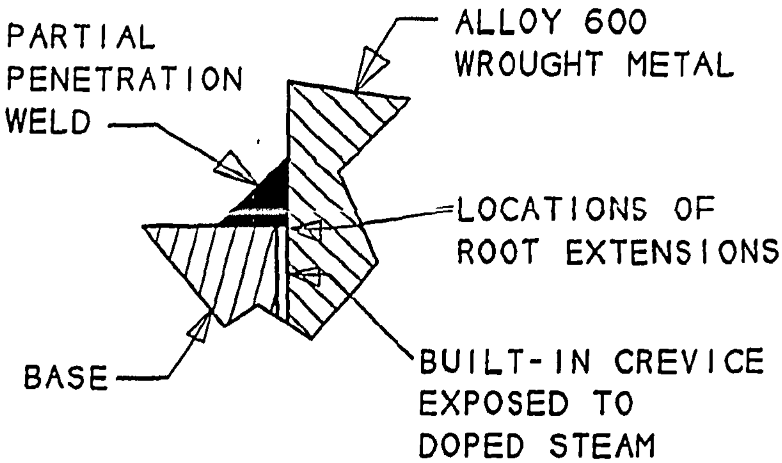

FIGURE 1. PARTIAL PENETRATION WELD GEOMETRY

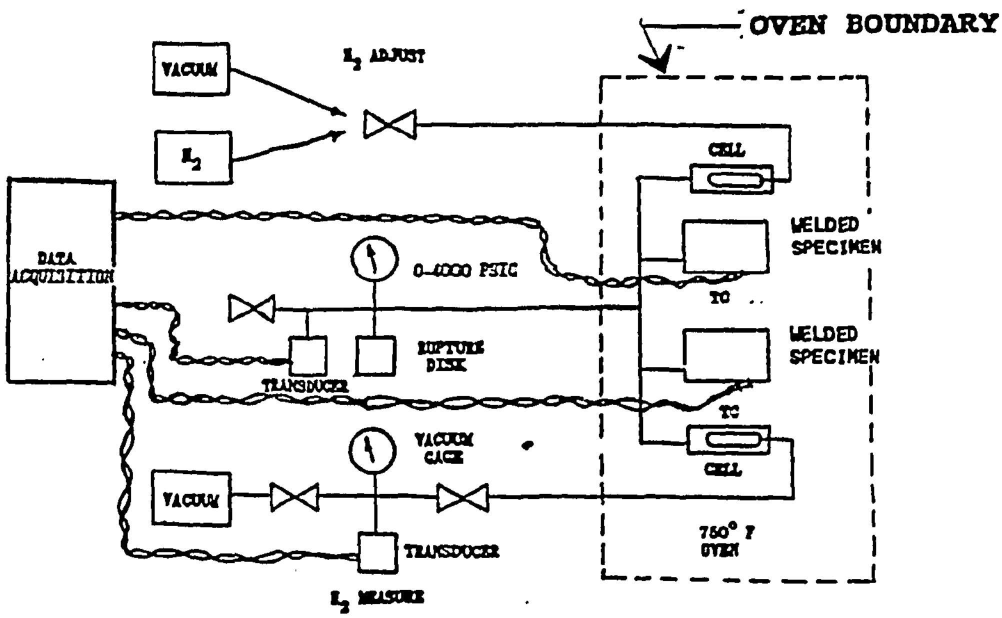

FIGURE 2. SCHEMATIC OF DOPED STEAM TEST
(Note: This figure will be redrawn for final copy)

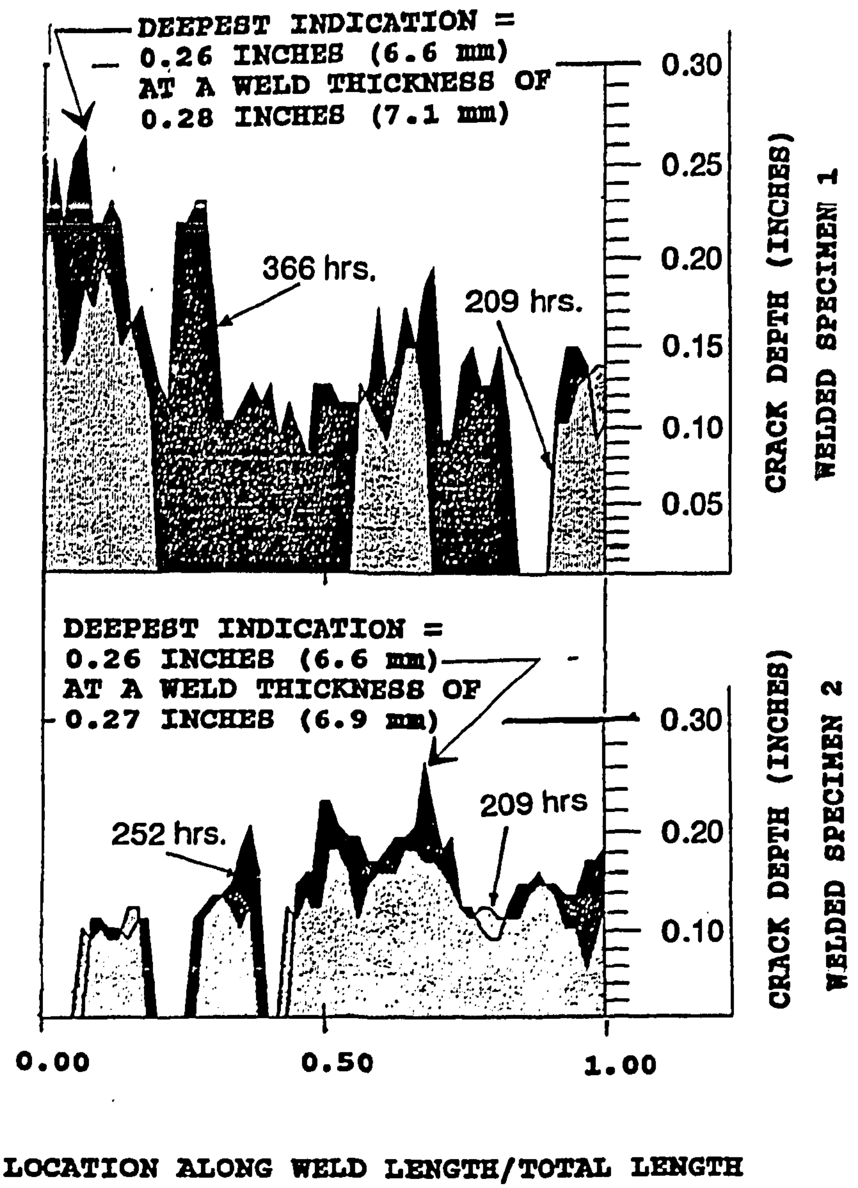

FIGURE 3. UT INDICATION OF CRACK SIZES IN WELD METAL OF WELDED SPECIMENS 1 AND 2 (1 INCH = 25.4 mm)

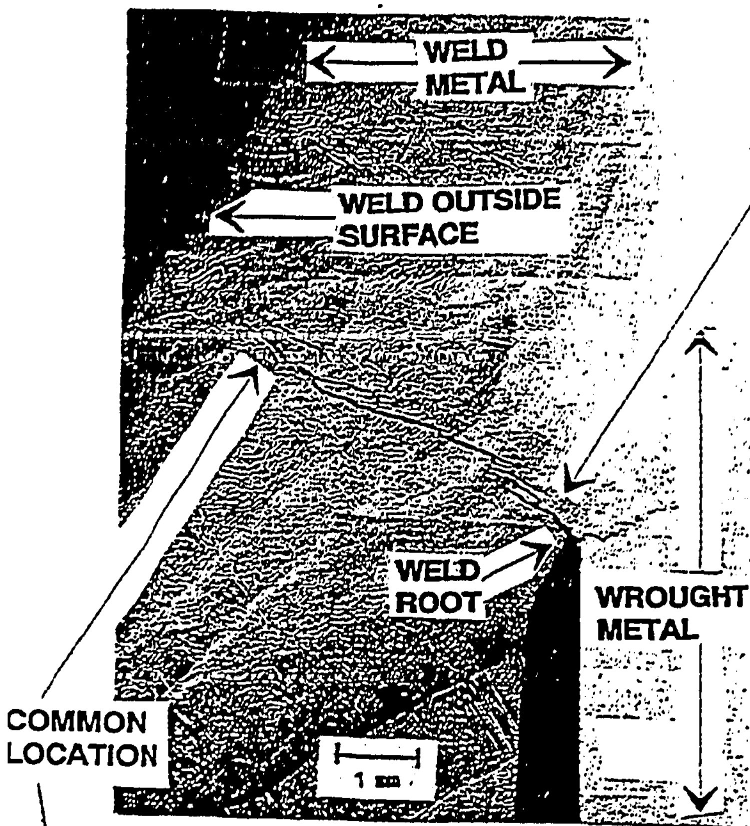

FIGURE 4a. CRACK OVERVIEW

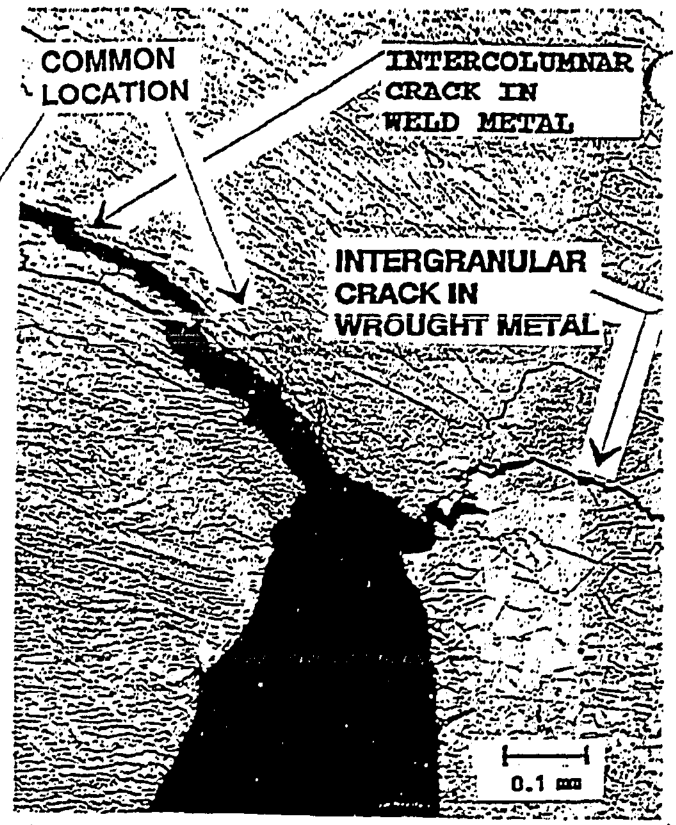

FIGURE 4b. WELD ROOT

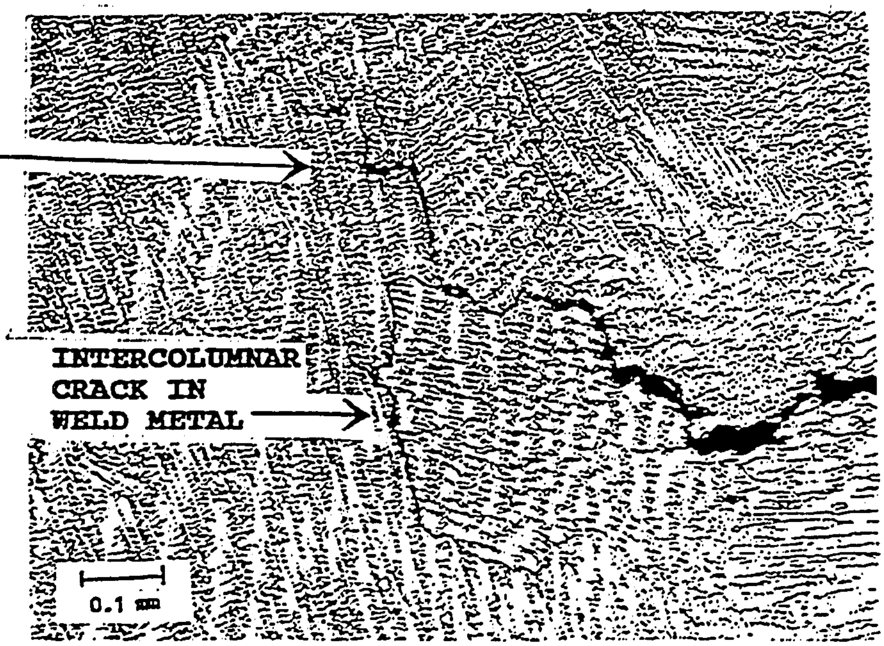

FIGURE 4c. CRACK TIP IN WELD METAL

FIGURE 4. PHOTOMICROGRAPHS OF WELDED SPECIMEN 2
AFTER 252 HOURS OF EXPOSURE TO
DOPED STEAM (PHOSPHORIC ETCH)

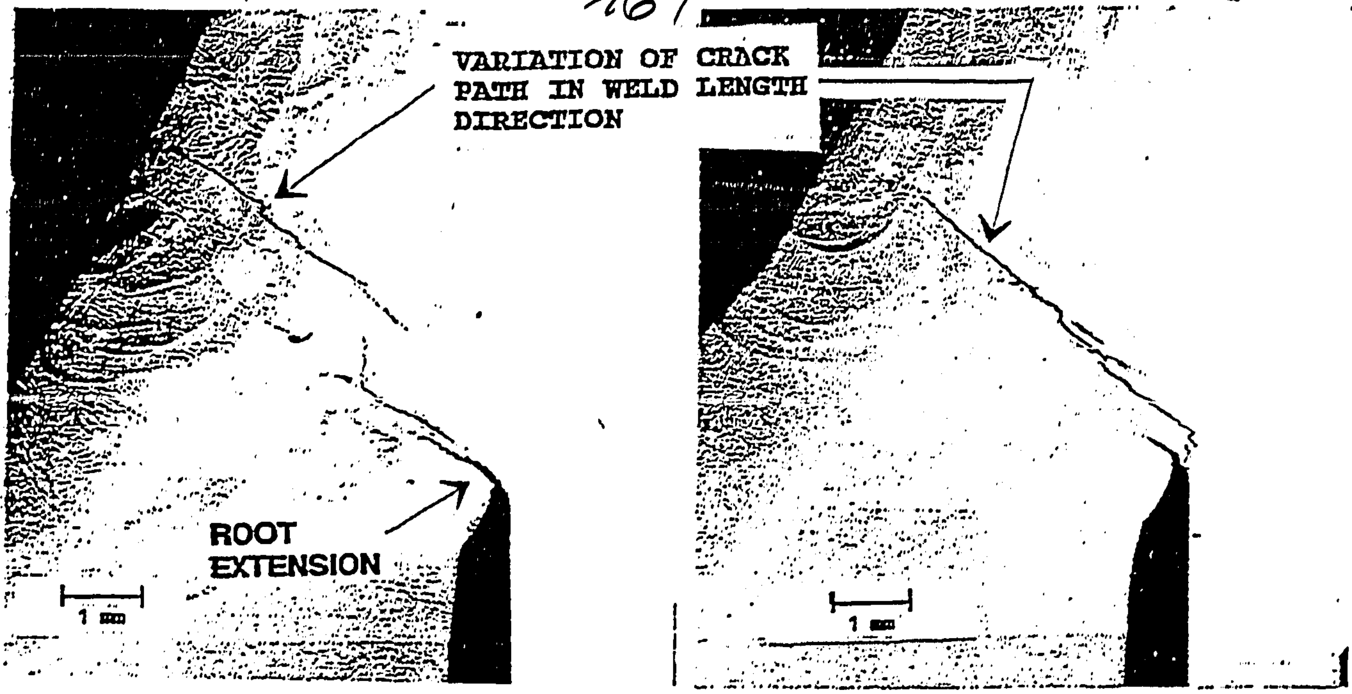

FIGURE 5a. PLANE 1

FIGURE 5b. PLANE 2

FIGURE 5. PHOTOMICROGRAPHS AT TWO PLANES OF POLISH SPACED 0.050 INCHES (1.27 mm) APART

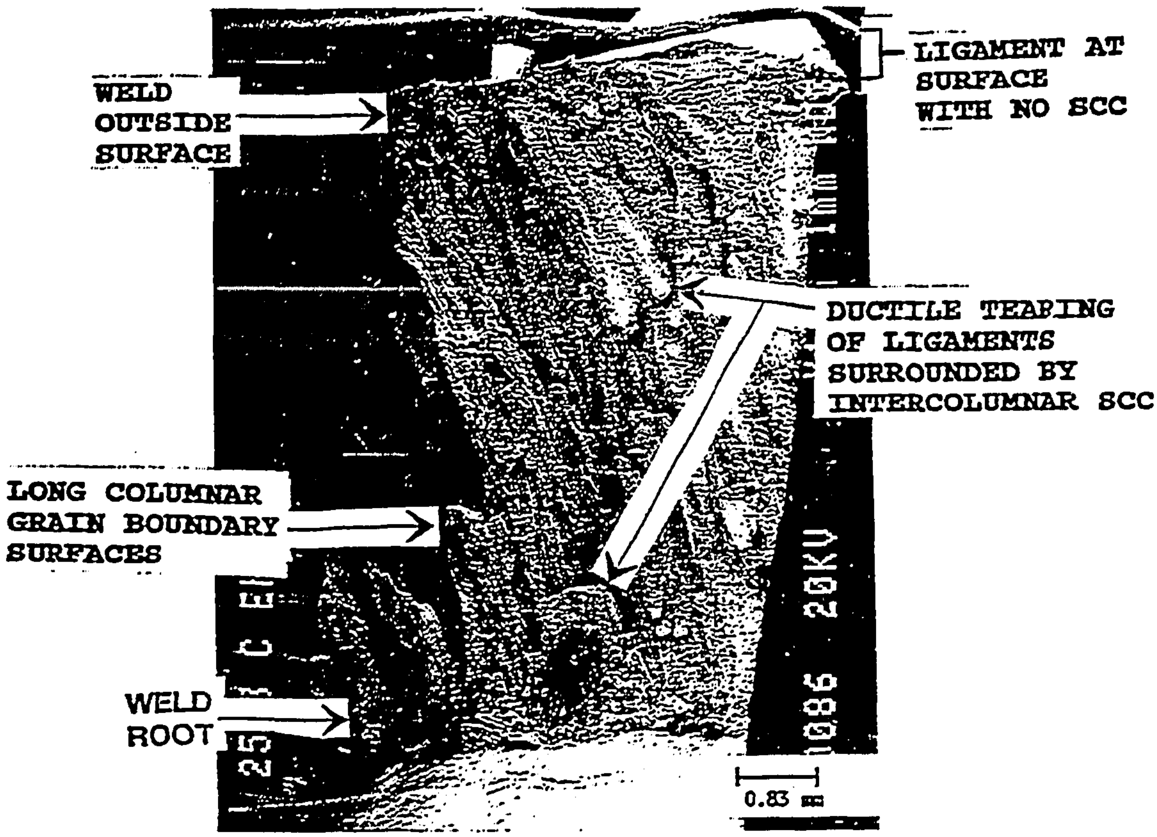

FIGURE 6. FRACTOGRAPH OF WELDED SPECIMEN 2