# EFFECTS OF OVERLOAD ON HYDROGEN SULPHIDE CRACKING

O. Jonas\*

## INTRODUCTION

Knowledge of effects of load or stress history on subsequent crack behaviour is needed for service and failure analysis and for testing of stress corrosion, corrosion fatigue and fatigue crack growth. Most of the structures, pressure vessels and machinery experience stress variations. In some stress corrosion testing of precracked specimens, the initial stress intensity is produced by bolt or wedge loading in air prior to placing the specimens into a test environment. In several applications, knowledge is needed of effects of overspeed test on stress corrosion properties of low alloy steels, particularly on the  $\kappa_{\rm ISCC}$  Plane-Strain  $\kappa_{\rm I}$ Threshold. While number of investigators reported data on fatigue crack growth delay after overload, for example [1 - 5], little information is available on corrosion fatigue [6] and stress corrosion [7, 8]. Effects of prestressing on KISCC of AISI 4340 and maraging 300 steels in R.T. 3.5% NaCl solution in H2O are reported in [7]. Behaviour of two maraging steels in dry R.T. hydrogen gas after overload in air is described in [8], where some fractographic observations are also included. All the above steels are high strength steels and a question arises about applicability of the results to low and medium strength steels. In this paper, the influence of overload in air on the sustained load cracking behaviour of forged low alloy medium strength bainitic steel in dry hydrogen sulphide is presented. The hydrogen sulphide environment was selected as a fast stress corrodant which gives similar KISCC values as some other more realistic service environments, but gives much shorter incubation time (time for a fatigue pre-crack to start propagating by stress corrosion) [9].

#### EXPERIMENTAL

# Material and Specimens

The IT-WOL specimens used in the programme were machined from a Ni Cr Mo V low alloy steel forging. All specimens were oriented with the notch and precrack plane in the radial-axial plane of a forged disk. Yield strength of the material is 1060 MPa (154 KSI), structure is tempered bainite, produced by austenitizing, water quenching and tempering at 600°C for 10 hours. Grain size is ASTM 5.

## Test Environment

Matheson C. P. grade hydrogen sulphide  $(H_2S)$  was used as the test environment. The gas has a minimum purity of 99.0 percent. A specially designed environment chamber was used, where two halves of the chamber are

<sup>\*</sup>Westinghouse Electric Corporation, Steam Turbine Division, Philadelphia, Pa., U.S.A.

clamped to the specimen. Containment for the environment was achieved by means of the O-ring seals in contact with the specimen faces and with an O-ring segment inserted into the machined notch of the test specimen. The test gas was admitted into the environment chamber following a purging procedure which consisted of heating the specimen-environment chamber assembly, and the associated inlet and discharge lines for 4 hours at about 360 K with dehumidified nitrogen flowing through the system. Cooling to the desired test temperature; and the introduction of the flowing test gas  $(\mathrm{H_2S})$ .

## Test Procedure

All specimens were fatigue precracked in air to a final stress intensity of 14.5 MPa·m<sup>1/2</sup> at a stress ratio R = 0.1 (R = minimum load/maximum load). The final precrack was longer than 3.8 mm from the tip of the machined starter notch. Immediately following the fatigue precracking, an overload was applied in air and the specimens were unloaded. The environment chamber was then attached to the specimen, and the system was purged and "baked out" in dehumidified nitrogen. Further testing was carried out in an Instron test machine. Hydrogen sulphide was allowed to flow through the environment system for at least 30 minutes before load application. The specimen was loaded at first to the lowest anticipated value for the apparent KISCC at a cross head speed of 30 mm/s. Load was maintained for at least 2 hours. If no crack growth took place during this period, load was increased to the next K level at the same cross-head speed and was held for at least 2 hours. This procedure was followed until cracking occurred. Once crack growth was initiated, it resulted in rapid growth and termination of the test.

## TEST RESULTS

Prior to the testing of overload effects,  $K_{\rm ISCC}$  in R.T. 20.6 to 27.5 kPa pressure hydrogen sulphide was established. It is about 18.6 MPa·m<sup>1/2</sup>. The results of overload testing are summarized in Table 1 and in Figure 1. Modest increases in the apparent  $K_{\rm ISCC}$  were observed after higher overloads. They are represented in the Figure 1 by a lower-bound line. In one experiment (specimen 15), after establishing "no growth" at  $K_{\rm I} = 27.4$  MPa·m<sup>1/2</sup> for 104 hours, the specimen was subjected to low amplitude cyclic loading ( $K_{\rm I}$  ranging from 24.5 to 27.3 MPa·m<sup>1/2</sup>) at 5 Hz. Rapid crack growth occurred following less than 400 cycles of fatigue. This suggests an elimination of the overload effect that increased apparent  $K_{\rm ISCC}$  from 18.6 to 27.4 MPa·m<sup>1/2</sup>.

# FRACTOGRAPHY

The fracture morphology was studied on most of the specimens by metallographic techniques and scanning electron microscopy (SEM). In addition, different yield strength specimens were loaded and unloaded in air and sectioned to see the crack front behaviour prior to stress corrosion cracking. Sides and mid-thickness of each specimen were investigated.

A typical morphology of the overload area with the subsequent stress corrosion crack is in Figure 2. It shows one half of the broken specimen No. 86 with a step (half branch) and/or branching at the overload area and the subsequent stress corrosion intergranular fracture. The angle between the original two branches varies from 90° to 180° and is appar-

ently influenced by the grain structure. Additional branching is visible in the stress corrosion area. Figure 3 shows crack branching after overload without stress corrosion, both on the specimen surface and in the mid-thickness. Figure 4 is a SEM picture of overloaded low yield strength (annealed) material, where the crack front branching phenomenon is exaggerated. The angle between two branches of the overload cracks is  $\sim 110^{\circ}$ .

#### DISCUSSION

The test results and fractographic observations indicate that an overload in air can increase the apparent K<sub>ISCC</sub> of the steel tested. The general behaviour and fractography is similar to that of maraging steels in hydrogen, overloaded in argon [8]. Compilation of available overload data for comparison is in Figure 5. Schematic representation and interpretation of the crack front deformation is in Figure 6. Crack branching, plastic deformation, and resulting compressive stresses seem to be responsible for the apparent increase of KISCC. The crack branching seems to be the dominating factor which diverts subsequent stress corrosion crack propagation from a straight direction. The branching during applied overloads was microscopic and is on the fractured specimens overshadowed by the subsequent stress corrosion crack growth. Quantitative explanation of the KISCC increase by crack branching is not possible because there are not enough measurements of branched cracks available. In trying to make a theoretical estimate of stress intensity changes with crack microbranching, we did not find any applicable source of analytical solutions. Solutions presented in [10] are not realistic in the region of microbranching because they do not connect to straight crack solutions for zero branching. Furthermore there are effects, such as anisotropy, transition from transgranular to intergranular cracking and transition from plastic zone to zero plasticity stress corrosion crack, which make microscopic and analytical explanations of the overload effects difficult.

#### CONCLUSIONS

- 1. Prior overload in air can cause an apparent increase of  $K_{\rm ISCC}$  of a low alloy medium strength bainitic steel. The increased  $K_{\rm ISCC}$  is approximately one half of an overload, with an exception of small overloads which are not effective.
- 2. Specimen loading in air has no effect on KISCC.
- 3. Fractography of tested specimens indicates crack front branching at the end of precrack (transgranular) and during the subsequent stress corrosion crack propagation (intergranular).

#### ACKNOWLEDGEMENTS

Most of the stress corrosion testing was done by Dr. R. P. Wei and Dr. T. T. Shih at Lehigh University.

# REFERENCES

 McMILLAN, J. C. and PELLOUX, M. N., "Fatigue Crack Propagation under Program and Random Loads", ASTM STP 415, 1968.

- 2. SHIJVE, J., Eng. Fracture Mech., 5, 1973, 269.
- 3. JONAS, O. and WEI, R. P., Int. J. of Fracture Mech., 7, 1971, 116.
- 4. HUDSON, C. M. and RAJU, K. N., "Investigation of Fatigue-Crack Growth under Simple Variable-Amplitude Loading", NASA TN D-5702, 1970.
- 5. KLESNIL, M. and LUKÁŠ, P., Eng. Fracture Mech., 4, 1972, 77.
- o. SHIH, T. T. and WEI, R. P., Proceedings of Intermatl. Conf. on Prospects of Fracture Mechanics, Noorthoff Publ., Leyden, 1974, 231.
- 7. CARTER, C. S., Report No. D6-25275, Boeing Co., 1970.
- 8. JONAS, O., Corrosion, 29, 1973, 299.
- 9. McINTYRE, P. and PRIEST, A. H., "Accelerated Test Technique for the Determination of KISCC in Steels", British Steel Corp., Report MG/31/72, 1972.
- 10. KITAGAVA, H., YUUKI, R. and OHIRA, T., Eng. Fracture Mech., 7, 1975, 515.

Table 1  $\,\,$  Influence of Prior Overload on Apparent  $\,\mathrm{K}_{\mathrm{ISCC}}$ 

Specimen No.	Overload K	K	Threshold Test Time at Load	Remark	Apparent K <sub>ISCC</sub> (Estimated)
5	32.0 (29.1)	18.7 (17.0) 22.0 (20.0)	2 hr. 2 min.	No growth Rapid growth	>18.7 (>17)
7	40.5 (36.9)	23.1 (21.0) 27.4 (24.9)	100 hr. 0.5 min.	No growth Rapid growth	>23.0 (>21)
15	65.9 (60.0)	27.6 (25.1) Fatigued at (22.5 to 25.0) 24.7 to 27.5	104 hr. (~400 cycles at 5Hz)	No growth	>27.5 (>25)
87	66.3 (60.3)	32.5 (29.6)	10 sec.	Rapid growth	<33.0 (<30)
4	68.0 (61.9)	22.0 (20.0) 27.5 (25.0) 33.0 (30.0) 38.5 (35.0) 43.8 (39.9)	2 hr. 2 hr. 2 hr. 2 hr. 10 sec.	No growth No growth No growth No growth Rapid growth	>38.5 (>35)
32	91.0 (82.8)	33.0 (30.0) 39.1 (35.6)	2 hr. 10 sec.	No growth Rapid growth	>33.0 (>30)
86	108.5 (98.7)	33.0 (30.0) 38.5 (35.0) 50.5 (46.0)	2 hr. 2 hr. 10 sec.	No growth No growth Rapid growth	>38.5 (>35)

- Notes: 1. K values are in MPa·m<sup>1/2</sup> (values in parenthesis are in ksi $\sqrt{\text{in}}$ ).
  - 2. All specimens fatigue precracked to a final  $K_{\rm max}$  of 14.5 (13.2), and preloaded in air to the indicated overload level.

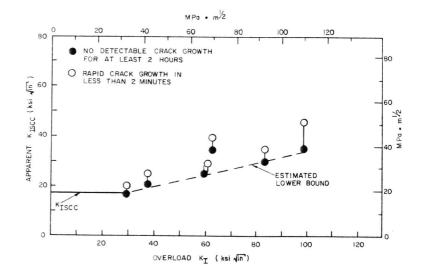


Figure 1 Apparent  $K_{\mbox{ISCC}}$  versus Overload  $K_{\mbox{I}}$ 

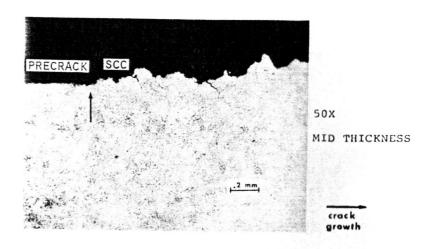
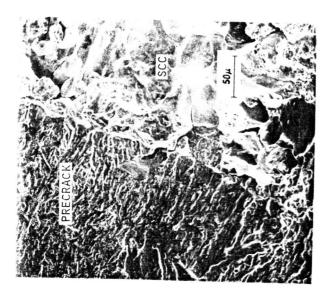


Figure 2 A Typical Fracture Morphology of the Overload Zone with the Subsequent Intergranular Stress Corrosion, Specimen 86 (continued)



300 X

specimen side A Typical Fracture Morphology of the Overload Zone with the Subsequent Intergranular Specimen 86

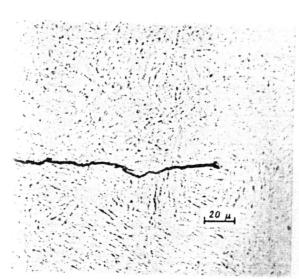
7



SIDE, 100 X



SIDE, 4000 X



MID THICKNESS, 500 X

Figure 3 Crack Front Branching After Overload and Unloading

274

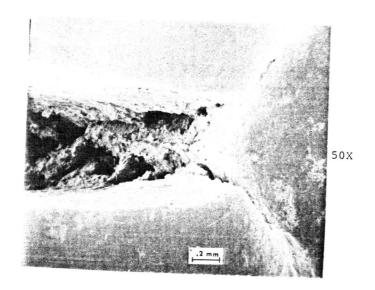


Figure 4 Exaggerated Branching after Static Load Application in Annealed Material

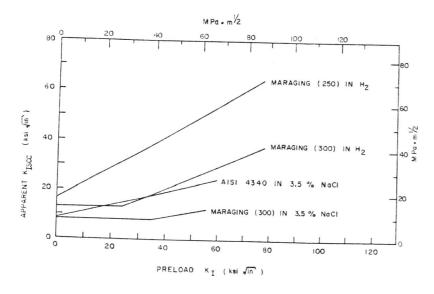


Figure 5 Apparent  ${\rm K}_{\rm ISCC}$  versus Preload  ${\rm K}_{\rm I}$  - Literature Data

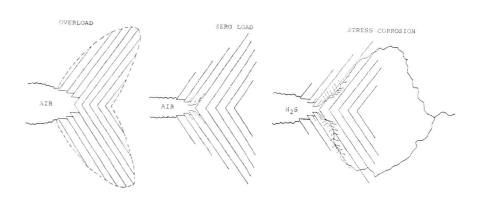


Figure 6 Schematic Representation of Crack Tip Deformation During and After Overload