

LOW CYCLE CORROSION FATIGUE OF AN Al-Zn-Mg-(Zr) ALLOY

Fu-Shiong Lin and E. A. Starke, Jr.\*

INTRODUCTION

There is ample evidence that the mechanical properties and deformation behaviour of materials can be influenced by altering the state of the surface [1 - 3]; however, considerable controversy [4 - 6] exists over the role of the surface layer in cyclic-hardening behaviour. This paper is concerned with the cyclic stress-strain response of an Al-Zn-Mg-(Zr) alloy in dry argon and in a corrosive solution of distilled water and H<sub>2</sub>SO<sub>4</sub> (pH = 2.7). The objective of this research was to evaluate the effect of the oxide layer on the fatigue behaviour in order to obtain information on the surface and bulk contributions to crack initiation and cyclic hardening. The oxide layer can have two major effects on the deformation characteristics: (a) adherent oxide films can act as barriers, preventing the egression of dislocations at the surface [1, 7], and (b) the high shear modulus of the oxide (the elastic constants of aluminum oxide are three to four times larger than those of aluminum [8]) produces an elastic repulsive force on dislocations in the substrate [2] which effects the hardening characteristics of the surface region.

EXPERIMENTAL PROCEDURES

The alloy used in this research was prepared at the Alcoa Technical Centre and received in as-hot-rolled plate form, with a nominal thickness of 0.58 cm. The composition in weight percent was 6.45% Zn, 2.08% Mg, 0.11% Zr, 0.05% Si, 0.04% Fe, balance aluminum. The microstructure after solutionizing at 480°C for 1 1/2 hrs was partially recrystallized (above 80%) and typical of that of commercially processed high strength aluminum alloys, Figure 1.

Fatigue samples were machined from the as-received plate with axes parallel to the rolling direction. The specimens were smooth and cylindrical in the gage section which was approximately 5 mm long by 3 mm in diameter. Multiple-sample, push-pull low-cycle-fatigue tests, described in detail elsewhere [9], were performed using strain control and by cycling about zero mean stress. Five different aged conditions, chosen from previous studies [10], were tested: (a) 4 hrs @ 120°C, (b) 2 hrs @ 120°C + 12 hrs @ 150°C, (c) 12 hrs @ 120°C + 12 hrs @ 150°C, (d) 24 hrs @ 120°C + 24 hrs @ 150°C, and (e) 24 hrs @ 150°C. The precipitate structure and monotonic deformation behaviour for these treatments have been presented elsewhere [10].

Two different environments were used for testing; dry argon and a distilled water-H<sub>2</sub>SO<sub>4</sub> solution with pH = 2.7. The latter was selected for its uniform corrosion of the aluminum alloy used, regardless of the aged condition. The corrosion rate was calculated by weight loss from corrosion

\*Georgia Institute of Technology, Atlanta, Georgia, U.S.A.

tests and was equivalent to  $4.2-4.6 \times 10^{-10}$  m/min, being independent of test time for tests as long as three days. The sample gage section was completely submerged in the solution during the corrosion LCF studies, and an elapsed time of 30 minutes was allowed from initial submersion to the beginning of the fatigue test. This time was sufficient to remove the  $50-100 \times 10^{-10}$  m thick oxide film normally present on aluminum and its alloys [11].

## RESULTS AND DISCUSSION

### Cyclic Hardening Behaviour

Based on the calculation of Livesay and Starke [2] the aluminum oxide, when present, should exert a very large repulsive force on the dislocations in the aluminum alloy substrate. This increases the probability for surface layer hardening as proposed by Kramer [12, 13] and Kramer and Kumar [4, 14]. The probability of surface layer hardening should be reduced when the oxide is absent, e.g. in the corrosive environment. The cyclic stress-strain response, represented as stress amplitude versus cycles, for samples aged 4 hrs @ 120°C and 24 hrs @ 150°C and tested in both dry argon and the H<sub>2</sub>O-H<sub>2</sub>SO<sub>4</sub> solution is shown in Figure 2. There is no significant difference in the cyclic hardening behaviour for the two environments, suggesting that the surface layer effect is not significant for these test conditions. These results are consistent with those of Finney and Laird [5] and Chen and Starke [6], who showed that cyclic hardening in single crystals was a bulk hardening effect, insensitive to the surface condition, and not controlled by the surface layer.

### Low Cycle Corrosion Fatigue

The results of the low cycle fatigue measurements are summarized in Table 1 and presented as  $\log \Delta \epsilon_p / 2$  versus  $\log 2N_f$  (Coffin-Manson relationship) in Figure 3. The slopes of the Coffin-Manson plots,  $c$ , were approximately the same for similarly aged samples tested in two environments, with the exception of those aged 24 hrs @ 150°C, and ranged between -0.60 and -0.73. The discontinuity observed for the argon data was absent for the H<sub>2</sub>O-H<sub>2</sub>SO<sub>4</sub> data, and this absence may be attributed to the much earlier failure in the corrosive medium. The cause of the discontinuity has been discussed elsewhere [9, 10].

The effect of aging on the low cycle fatigue behaviour was independent of environment. The poorest fatigue resistance was shown by samples aged 4 hrs @ 120°C, a treatment that produced small Guinier-Preston zones which are sheared by dislocations resulting in a banded deformation structure [9, 10]. The best fatigue resistance for the range of strain amplitudes tested was shown by samples aged 24 hrs @ 150°C, a treatment which produced semi- and incoherent MgZn<sub>2</sub> particles which are looped by dislocations producing a more homogeneous deformation than those of the other aging treatments of this study [10]. A reduction in life by one half occurred when testing in the corrosive environment. An examination of the ratio, cycles to crack initiation/cycles to failure, revealed that the liquid environment was equally detrimental to crack initiation and crack propagation. The ratio varied, depending on the magnitude of the plastic strain amplitude, from 0.67 to 0.88, and was insensitive to environment. Optical examination of the surface at various stages of testing revealed that the cracks initiated, in order of frequency, (a) at slip bands in the *wire-crystallized* grains, (b) at pits and inclusions, and (c) along grain boundaries of recrystallized grains which were approximately normal to the

stress direction. The removal of the oxide film by the corrosive environment increases the probability of dislocation egression from the surface and accelerates extrusion-intrusion formation and crack initiation. In addition, crack initiation may be enhanced by preferential dissolution at deformation bands, inclusions and grain boundaries.

In order to establish the effect of environment on crack propagation, some samples were cycled in the H<sub>2</sub>O-H<sub>2</sub>SO<sub>4</sub> solution until the crack had propagated approximately one quarter way through the cross section. The liquid was then removed and the sample cycled to failure in dry argon. The fracture surfaces were examined in the scanning electron microscope and representative areas are shown in Figure 4. The type and spacing of the fatigue striation are environment sensitive. When crack propagation occurred in the argon environment, closely spaced, ductile striations were formed. However, brittle striations having "cliff edges" and "river markings" running normal to the striations were formed during crack propagation in the H<sub>2</sub>O-H<sub>2</sub>SO<sub>4</sub> solution. Similar striations have been observed by Stubbington [15] on aluminum samples fatigued to failure in a NaCl solution. The mechanism for changing the fracture mode may be due to anodic dissolution at the crack tip [16] and to the formation and adsorption of hydrogen gas by cathodic reactions [17, 18] in this region.

## CONCLUSIONS

The cyclic hardening observed in this study is a result of a bulk hardening effect and is insensitive to the surface condition and not controlled by the surface layer. Both crack initiation and propagation are environment sensitive; initiation being effected by the corrosive environment's influence on intrusion-extrusion formation, and propagation being effected by the corrosive environment producing a change in fracture mode from ductile to brittle.

## ACKNOWLEDGEMENTS

This research was sponsored by the Air Force Office of Scientific Research, Air Force Systems Command, USAF, under Grant No. AFOSR-74-2615, Dr. Alan H. Rosenstein, Program Manager. The United States Government is authorized to reproduce and distribute reprints for Government purposes notwithstanding any copyright notation here on.

## REFERENCES

1. BARRETT, C. S., Acta Met., 1, 1953, 2.
2. LIVESAY, B. R. and STARKE, E. A., Jr., Acta Met., 21, 1973, 247.
3. NAKAGOWA, Y. G. and GREENFIELD, A. G., Acta Met., 21, 1973, 367.
4. KRAMER, I. R. and KUMAR, A., "Corrosion Fatigue", eds. O. J. Devereux, A. J. McEvily and R. W. Staley, NACE-2, Houston, Tex., 1972, 146.
5. FINNEY, J. M. and LAIRD, C., Phil. Mag., 31, 1975, 339.
6. CHEN, E. Y. and STARKE, E. A., Jr., Mater. Sci. Eng., 24, 1976, 209.
7. WESTWOOD, A. K. C., Phil. Mag., 5, 1960, 981.
8. HEAD, A. K., Phil. Mag., 44, 1953, 92.
9. SANDERS, T. H., Jr. and STARKE, E. A., Jr., Met. Trans., 7A, 1976.
10. SANDERS, R. E., Jr. and STARKE, E. A., Jr., Submitted to Mater. Sci. Eng.
11. Aluminum, 1, ed. K. R. Van Horn, ASM, 1967, 209.

12. KRAMER, I. R., Trans. ASM, 62, 1967, 209.
13. KRAMER, I. R., Proc. Air Force Conf. on Fatigue and Fracture of Aircraft Structures and Materials, Miami Beach, 1969, 271.
14. KRAMER, I. R. and KUMAR, A., Met. Trans., 3, 1972, 1223.
15. STUBBINGTON, C. A., Metallurgia, 68, 1963, 109.
16. UHLIG, H. H., "Corrosion Fatigue", The Univ. of Conn., 1972, 270.
17. WEI, R. P., Inter. J. Fract. Mech., 4, 1968, 159.
18. BRADSHAW, F. J. and WHEELER, C., Appl. Mat. Res., 5, 1966, 112.

Table 1 Low Cycle Fatigue Data of Al-Zn-Mg-Zr Alloy After Various Aging Treatments

Sample	Yield (MN/m <sup>2</sup> )		-c***		e' f (%)****		cycles for initiation (Avg.)		cycles for failure	
	Argon*	H <sub>2</sub> O-H <sub>2</sub> SO <sub>4</sub> **	Argon	H <sub>2</sub> O-H <sub>2</sub> SO <sub>4</sub>	Argon	H <sub>2</sub> O-H <sub>2</sub> SO <sub>4</sub>	Argon	H <sub>2</sub> O-H <sub>2</sub> SO <sub>4</sub>	Argon	H <sub>2</sub> O-H <sub>2</sub> SO <sub>4</sub>
	4 @ 120	368	--	0.62	0.60	43	34	.82	.82	
DA 212	445	--	0.71	0.68	98	43	.78	.80		
DA 1212	442	--	0.69	0.65	69	50	.77	.78		
DA 242	415	--	0.70	0.70	116	80	.80	.84		
24 @ 150	378	--	0.48	0.73	18	86	.77	.77		
			0.92		632					

\* Data of Sanders and Starke [10]  
 \*\* No tensile tests were conducted in this environment  
 \*\*\* Fatigue ductility exponent  
 \*\*\*\* Fatigue ductility coefficient

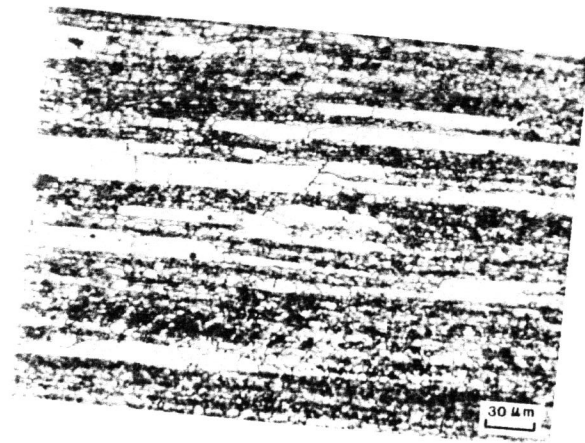


Figure 1 Photomicrograph of Longitudinal Section of the Solutionized Al-Zn-Mg-Zr Plate. Kellers etch

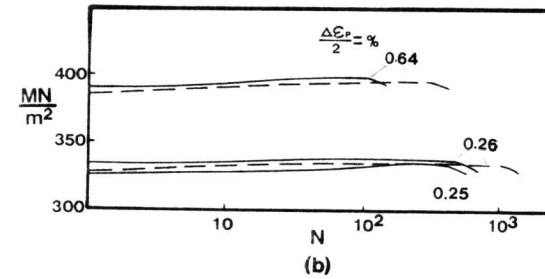
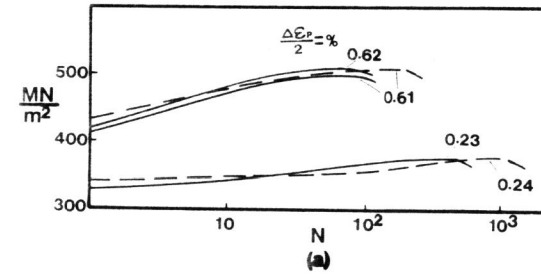


Figure 2 Stress Amplitude versus Number of Cycles

(a) Specimens Aged 4 Hours at 120° C  
 (b) Specimens Aged 24 Hours at 150° C

Solid Lines Represent Tests in H<sub>2</sub>O-H<sub>2</sub>SO<sub>4</sub> Solution; Dashed Lines Represent Tests in Dry Argon

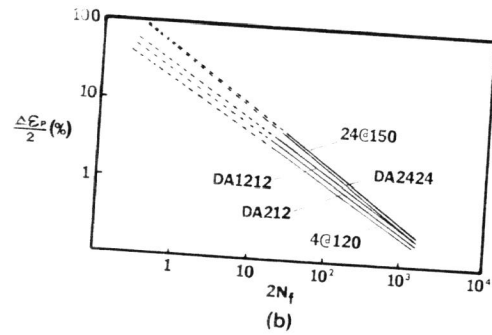
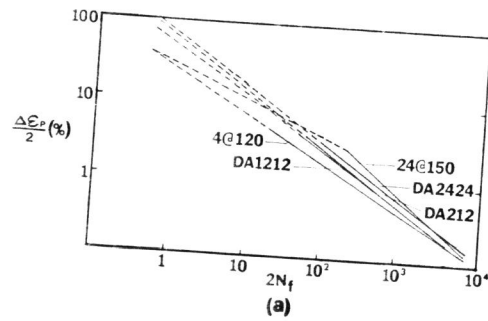
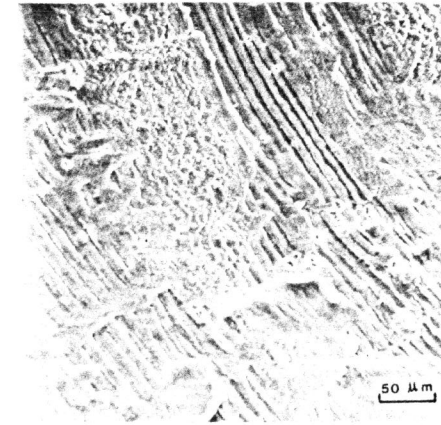
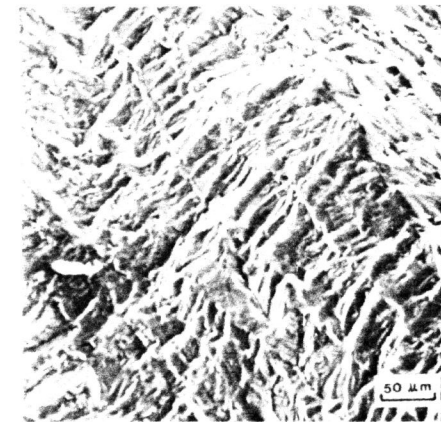


Figure 3 Effect of Aging Treatment on Low Cycle Fatigue Life  
 (a) Specimens Fatigued in Dry Argon [10]  
 (b) Specimens Fatigued in H<sub>2</sub>O-H<sub>2</sub>SO<sub>4</sub> Solution



(a)



(b)

Figure 4 Scanning Electron Micrograph of LCF Fracture Surfaces  
 (a) Crack Propagated in Dry Argon  
 (b) Crack Propagated in H<sub>2</sub>O-H<sub>2</sub>SO<sub>4</sub> Solution