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Estimation of environment-induced crack growth rate as a function of stress intensity factors generated during slow strain rate testing of aluminum alloys

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Abstract:

In this contribution we introduce a simple approach to quickly estimate the environment-induced crack velocity (CV) as a function of the calculated applied stress intensity factor (K) developed during the slow strain rate testing of aluminum alloys exposed to aqueous or humid-air type environments. The CV-K behavior for a commercial aluminum-magnesium alloy, AA5083-H131, sensitized and pre-exposed to a 0.6M NaCl solution has been estimated from slow strain rate test data. The predicted threshold K and crack velocities match recently published data for the same alloy in similarly sensitized conditions where the CV-K data was obtained using state-of-the-art fracture mechanics based testing.

Keywords: aluminum alloys; corrosion; fracture; stress corrosion cracking

1 Introduction

Environment-induced cracking (EIC) in commercial higher strength aluminum alloys (Al-Zn-Mg-Cu, 7xxx series and Al-Cu-Mg, 2000 series) has been assessed in mechanically loaded pre-cracked test specimens exposed to various environments since the late 1960's (Mulherin, 1967; McEvily et al., 1967; Hyatt, 1969). Early findings from these bolt-loaded double cantilever beam (DCB) test specimens (Hyatt, 1969) are summarized elsewhere, in terms of an estimated crack growth rate (CV) as a function of the applied stress intensity factor (K), commonly referred to as CV-K curves (Hyatt, 1969; Spiedel and Hyatt, 1972; Spiedel, 1975). Published data for Al-Zn-Mg-Cu, 7xxx series alloys has been recently reviewed in detailed elsewhere (Holroyd & Scamans, 2013).

Constant-displacement, static loading based test methods, such a bolt-loaded DCB testing, while providing experimentally simple and potentially relatively low-cost options are often compromised by a lack of reproducibility (Dietzel, 2000) and very long test times (e.g. 10,000 hr) to obtain threshold stress intensity factors, K_{ISCC} . In addition, experimental issues associated with out-of-plane crack growth or crack-branching (Domak, 1990) and/or crack corrosion

product wedging generating local stresses (Schra & Faber, 1981; McNaughtan et al., 2003) can potentially invalidate K_{1SCC} values associated with crack arrest under decreasing K .

Rising-displacement based tests methods, developed over the last 25 years or so, while potentially considerably shortening test times needed to generate threshold data have their own potential issues, including a) validity of output data is strongly dependent on the selection of an appropriate displacement rate (Dietzel, 2000; Dietzel & Mueller-Roos, 2001) which must be independently verified on a case-by-case basis, typically using fractographic evidence (Dietzel, 2000) and b) test methods require state-of-the-art fracture mechanics based testing equipment and thereby no longer offer a 'low-cost' test option.

Usage of CV-K data to formally assess/quantify the potential susceptibility of commercial aluminum alloys to EIC with respect to service performance remains limited, despite detailed evaluations of the approach (Sprowls et al., 1973 & 1976; Kaufman et al., 1976; Domack, 1990; Dietzel, 1999) and publication of Standardized Test Methods in the US (ASTM, 1995) and Europe (ISO, 1998).

We present a low-cost approach to quickly estimate the CV-K relationship for the EIC of high-strength aluminum alloys. The method relies upon the acquisition of high-quality (low-noise) nominal stress as a function of the extension (relative plastic strain) promoted during the slow strain rate testing (SSRT) of conventional round-bar tensile samples. Prior to straining the specimens are given a pre-exposure treatment creating corrosion features to provide EIC crack initiation sites, free of any significant associated local stresses and limited local plasticity, unlike those associated with either fatigue pre-cracks or mechanical 'pop-in' cracks from a machined notch, as are typically generated in test specimens used to generate CV-K curves.

2 Materials and Methods

2.1 Materials

The alloy selected for testing was a commercial Al-Mg-Mn alloy, AA5083-H131, containing (wt%) 4.46 Mg, 0.62 Mn, 0.12 Fe, 0.08Cr, 0.07 Si and 0.02 Zn (established using optical Emission Spectroscopy, a standard technique used in the aluminum industry for melt analysis of solidified samples) supplied in the form of 29-mm-thick rolled plate in the H131 condition. The alloy had a typical non-recrystallized fibrous microstructure with grain sizes of 150 μm , 80 μm , and 35 μm in the longitudinal (L), long-transverse (LT) and short-transverse (ST) directions, respectively (Seifi et al, 2016). A 0.2% yield strength of 270 MPa and ultimate tensile strength (UTS) of 375 MPa were obtained for the ST direction when tested in air at 1×10^{-3} /s, along with a K_{IC} of 31 $\text{MNm}^{-3/2}$.

For SSRT, smooth tensile specimens with a gauge length and diameter of 12.7 and 3.2 mm, respectively, were machined with their major axis parallel to the short-transverse direction in order to maximize susceptibility to EIC.

2.2 Sensitization, Pre-exposure and SSRT

Alloy sensitization was conducted at 80 °C in an air-oven. 250 h sensitization at 80 °C resulted in a NAML value of 25-30 mg/cm² (Seifi et al, 2016).

Ahead of pre-exposure to 0.6M NaCl, the tensile specimen gauge lengths were mechanically polished with 3/0 emery paper (soaked in paraffin oil) and ultrasonically degreased in acetone to ensure the generation of oil-free surfaces. After polishing, tensile samples were pre-exposed in 0.6M NaCl at RT under full-immersion conditions for times up to 375 hours.

Immediately following the pre-exposure, SSRT tensile specimens were given a gentle rinse using a small volume of distilled water (DW), carefully dried with compressed air and within two minutes subjected to SSRT employing a nominal strain rate of $\sim 10^{-5}$ /s. The samples were exposed to either humid laboratory air (relative humidity, 50%) or dry air, achieved by packing the test specimen in anhydrous magnesium perchlorate granules (Willard & Smith, 1922). Surface films produced during pre-exposure were not removed prior to SSRT.

Fractured SSRT test specimens were cleaned in ethanol followed by examination in a FEI Quanta Scanning Electron Microscope in order to characterize fracture surface details, identify and map the various fracture modes and measure the linear depths of intergranular corrosion (IGC) and environment-induced cracking.

2.3 Estimation of EIC growth rate/implied stress intensity factor during SSRT

The method to estimate EIC depth and growth rates and the applied stress intensity factors generated during SSRT for tensile samples with a given sensitization/pre-exposure history, comprised of the following steps:

- 1) Calculation of nominal stress differentials as a function of the relative plastic strain for SSRT conducted in humid and dry air for the samples subjected to the same sensitization and pre-exposure treatments,
- 2) Assuming the stress differentials from 1) are directly relatable to an implied loss of load-bearing area attributable to EIC, calculate the EIC area developed during SSRT in humid air using the relationship:

$$\text{Area of EIC} = \text{Original cross-sectional area} \times [1 - (\text{Stress in Humid Air})/(\text{Stress in Dry Air})] \quad \text{Equation 1}$$

where Area is in mm² and stress in MPa

- 3) Estimate EIC crack depths as a function of relative strain (or time) during SSRT for various crack shapes using the 'Area of EIC' and an assumption EIC is predominantly associated with a single crack,
- 4) Calculate stress intensity factor (K) for the various crack shapes as a function of time by inputting the relevant stresses (taken from the SSRT data) and estimated crack depths (from 3 above) into published relationships for K as a function of load and crack depth (Sprowls et al., 1984; Stark & Ibrahim, 1986, 1987; Carpinteri, 1992; Shih & Chen, 2002) for cylindrical samples containing surface cracks.
- 5) Generate EIC crack velocity – stress intensity factor dependences (CV-K curves), using information gleaned from 1) to 4).

3 Results

3.1 SSRT Data

Engineering stress as a function of the % relative plastic strain realized during SSRT conducted on as-received and sensitized AA5083-H131 samples strained in humid air (50% RH) and dry air at a nominal strain rate of 10^{-5} /s after pre-exposure to 0.6M NaCl are provided in Figure 1. The mechanical properties extracted, depths of intergranular corrosion (promoted during pre-exposure to 0.6M NaCl) and depths of EIC generated during SSRT reported earlier are shown in Table 1 and further detailed background information on this SSRT is provided elsewhere (Holroyd et al., 2017).

Table 1: SSRT data for AA5083-H131 subjected to SSRT at a nominal strain rate of 10^{-5} /s in either humid air (50% RH) or dry air.

Test #	Sensitization at 80 °C	Exposure in 0.6M NaCl (hr)	UTS (MPa)	YS (MPa)	IGC Depth (μm)	Type-1 EIC		SSRT Conditions
						K _{ISCC} (MNm ^{-3/2})	Depth (μm)	
4	None	375	329	257	65-90	3.8	500	Humid air
6	None	375	353	255	60	-	-	Dry air
7	250	250	273	237	110	4.2	890	Humid air
9	250	250	354	245	110	-	-	Dry air

Figure 1: Representative engineering stress versus relative plastic strain curves for as-received and sensitized AA5083-H131 after pre-exposure to 0.6M NaCl and then strained in either humid air (50% RH) or dry air at a nominal strain rate of 10^{-5} /s.

The relative extension data from the SSRT test's conducted in dry air (Test's #6 and #9) were shifted in the analysis so that the load – extension (time) curves during the later stages of elastic behavior overlap with equivalent test conducted in humid air (Test's #4 and #7), Figure 2.

Figure 2: Comparison of the stress levels achieved in humid (50% RH) and dry air as function of the imposed extension during slow strain testing after pre-exposure to 0.6 M NaCl. A) As-received AA5083-H131 + 375 hr in 0.6M NaCl and B) AA5083-H131 sensitized for 250 hr at 80 °C + 250 hr in 0.6M NaCl.

Stress level differentials as a function of the extension imposed for SSRT conducted in humid and dry air are calculated and based on an assumption these stress differentials are directly relatable to losses of load-bearing area (see Equation 1), they are used in the estimation of the implied area of EIC generated as a function of the % relative strain. Estimated areas of EIC generated during the SSRT of AA5083-H131 in humid air (50% RH) are presented in Figure 3, as a function of time following the imposed stress during straining exceeding around 200 MPa.

Figure 3: Estimated areas of EIC as a function of time during the SSRT of as-received (Test #4) and sensitized (Test #7) AA5083-H131 strained in humid air after pre-exposure to 0.6M NaCl.

Maximum EIC crack depths as a function of the cracked area can be estimated for various crack shapes, such as elliptical, chord or an external peripheral ring. Crack depths, are estimated assuming one main crack initiates from the external surface of the specimen with the results shown in Figure 4, where a 'half-penny' aspect ratio is assumed for an elliptical crack, as often is observed during the early stages of EIC (Holroyd & Hardie, 1982; Burnett et al., 2017).

Figure 4: Estimated EIC depths for different crack geometries developed during the SSRT of as-received and sensitized AA5083-H131 pre-exposed to 0.6M NaCl and strained in humid air at a nominal strain rate of 10^{-5} /s. A) As-received + 375 hr pre-exposure to 0.6M NaCl and B) Sensitized 250 hr 80 °C + 250 hr in 0.6M NaCl.

Having obtained the implied crack depths as a function of time for various crack shapes we can now calculate the associated crack growth rates as a function of time, Figure 5. Then using knowledge of the load applied during the SSRT as a function of time and assuming a crack profile we can calculate the local stress intensity factor, K, as a function of time, using published relationships for stressed round bars (Stark & Ibrahim, 1986, 1987; Carpinteri, 1992; Shih & Chen, 2002), Figure 6.

Figure 5: Predicted EIC growth rates for different crack geometries during the SSRT of as-received and sensitized AA5083-H131 pre-exposed to 0.6M NaCl and strained in humid air at a nominal strain rate of 10^{-5} /s. A) As-received + 375 hr pre-exposure to 0.6M NaCl and B) Sensitized 250 hr 80 °C + 250 hr in 0.6M NaCl.

Figure 6: Predicted stress intensity factors for different crack geometries associated with EIC during the SSRT of as-received and sensitized AA5083-H131 pre-exposed to 0.6M NaCl and strained in humid air at a nominal strain rate of 10^{-5} /s. A) As-received + 375 hr pre-exposure to 0.6M NaCl and B) Sensitized 250 hr 80 °C + 250 hr in 0.6M NaCl.

CV-K curves, Figure 7, are obtained using data provided in Figures 5 and 6.

Figure 7: Predicted CV-K behaviour (assuming different geometries) promoted during the SSRT of as-received and sensitized AA5083-H131 pre-exposed to 0.6M NaCl and strained in humid air at a nominal strain rate of 10^{-5} /s. A) As-received + 375 hr pre-exposure to 0.6M NaCl and B) Sensitized 250 hr 80 °C + 250 hr in 0.6M NaCl. CV-K curves from literature (A,B) provided for comparison.

4 Discussion

Subjecting AA5083-H131 tensile specimens to pre-exposure in 0.6 M NaCl ahead of SSRT led to simultaneous hydrogen pre-charging and localized intergranular corrosion (IGC) providing local stress raisers, arguably transforming the originally smooth tensile specimens into pseudo pre-cracked test specimens but with little associated plasticity. This enables EIC initiation in humid air at considerable higher nominal strain rates than those required without pre-exposure (Holroyd et al., 2017 a, b).

The agreement between maximum predicted EIC depths, obtained from analysis of load–extension data obtained during SSRT, Figure 8, and those observed on the fracture surfaces of failed SSRT tensile samples, Figure 9, for both the as-received and sensitized AA5083-H131 is remarkable, particularly considering our assumption to attribute loading-bearing area losses during SSRT to a single region of EIC, when its evident multiple EIC cracks have initiated during SSRT and several typically contribute to SSRT failures, Figure 9.

Figure 8: Predicted EIC depths for different geometries in AA5083 using load-extension data obtained during the SSRT of as-received and sensitized AA5083-H131 pre-exposed to 0.6M NaCl and strained in humid air at a nominal strain rate of 10^{-5} /s . A) As-received + 375 hr pre-exposure to 0.6M NaCl and B) Sensitized 250 hr 80°C + 250 hr in 0.6M NaCl.

Predicted EIC growth rates for the sensitized AA5083-H131 display a sudden increase when EIC depths exceed $\sim 580 \mu\text{m}$, Figure 8B, whereas no such increase was obvious for the as-received material, Figure 8A. It is interesting to note in harmony with these predictions, fracture mode evaluation during SSRT indicates the sensitized material underwent an EIC mode transition, from solely Type-1 EIC to a mixed mode involving Type-1 and Type-2 EIC, when EIC depths exceeded $\sim 580 \mu\text{m}$, Figure 9B, whereas limited Type-2 EIC was detected in as-received material.

A more detailed comparison of the predicted and experimentally observed EIC behavior promoted during our SSRT of AA5083-H131 in humid air after pre-exposure to 0.6M NaCl is provided in Table 2, from which it is apparent the predicted EIC depths are slightly lower than those observed experimentally. This difference we attribute to multiple EIC crack initiation and

crack coalescence during SSRT, the results of which are apparent in the fracture surfaces shown in Figure 9.

Despite these differences our initial findings indicate the accelerated test method presented here should be capable of quickly providing, an indication of an aluminum alloys EIC threshold stress intensity factor, K_{IEIC} , and its potential EIC susceptibility as a function of alloy sensitization/temper.

Table 2: Comparison of predicted and experimentally observed EIC characteristics promoted during the SSRT of as-received and sensitized AA5083-H131 in humid air (50% RH) after pre-exposure to 0.6M NaCl.

		Observed (Holroyd et al., 2000)	Predictions using SSRT data	
		Type 1-EIC	Type 1-EIC	Type-2 EIC
As-Received	Depth, μm	500	400	400 - 500
	CV (m/s)	---	$\sim 10^{-7}$	$> 10^{-7}$
	K ($\text{MN}^{-3/2}$)	3.8 - 11.3	$> 3 - 9$	> 9
Sensitized	Depth (μm)	890	580 580-700*	700 - > 1500
	CV (m/s)	---	$\sim 7 \times 10^{-7}$ $\sim 2.8 \times 10^{-6} *$	Numerous sudden jumps (Av. CV $\sim 2.8 \times 10^{-6}$)
	K ($\text{MN}^{-3/2}$)	4.2 – 12.4	3 – 8 8 – 11*	> 11.0

*Mixed mode Type-1 and Type-2 EIC

Figure 9: SEM images of fracture surfaces of SSRT samples strained to failure in humid air (50% RH) at a nominal strain rate of 10^{-5} /s. A) AA5083-H131 as-received + 375 hr in 0.6M NaCl (Test # 4C) A) AA5083-H131 + 250 hr 80 °C + 375 hr in 0.6M NaCl (Test #7); B) and D) are higher magnifications of A) and C) respectively.

Perhaps the most significant evidence for our assertion that the CV-K characteristics for AA5083-H131 may be acquired in this way is provided by:

a) A recent 3D Tomography time-lapse study on the SSRT of pre-exposed sensitized AA5083-H131 material used here (Gudla et al, 2019) which confirms Type-1 EIC initiation is typically associated with IGC stress raisers and occurs when stress levels exceed ~200 MPa with initial EIC growth predominantly evolving as a half-penny shaped elliptical cracked region and b) reasonable agreement exists between the predicted CV-K behaviour and that reported for AA5083-H131 in similar tempers obtained using state-of-the-art fracture mechanics based testing equipment (Steiner & Burns, 2018), Figure 7.

5 Conclusions

- 1) An accelerated test method which can provide crack growth rate (CV) as a function of the applied stress-intensity factor (K), (i.e. the CV-K behaviour) of EIC using slow strain rate testing is presented for as-received and sensitized AA5083-H131.
- 2) Predicted CV-K behaviour closely resembles published experimental data obtained using sophisticated fracture-mechanics based test methods.
- 3) The new approach employs a pre-exposure treatment ahead of straining to pre-charge tensile test specimens with hydrogen, while simultaneously introducing local intergranular corrosion sites to act as local stress raisers with limited associated local plasticity (i.e. 'chemical' pre-cracking) during SSRT.
- 4) Estimation of EIC areas promoted during SSRT as a function of time are calculated, assuming stress reductions between SSRT in humid and dry air after given plastic extension reflect a loss of load-bearing area, which is directly attributable to an area of EIC.
- 5) 3D time-lapse tomography studies of AA5083-H131 with identical sensitization, pre-exposure and SSRT conditions reveals confirms assumptions made on EIC initiation conditions and EIC growth for stress intensity factors up to around $15 \text{ MNm}^{-3/2}$ ($\sim 50 K_{IC}$).

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