

#### Joint AFRL – FAA Technical Interchange Meeting (TIM) on Environmentally Assisted Cracking (EAC) of High-Strength 7XXX Series Aluminum Alloys

Updated March 2025

#### Table of Contents

Hunter: AFRL Introduction	3		
Wertz: AFRL Materials and Manufacturing Directorate			
Gorelik: FAA Opening Remarks	15		
Waite: EASA Certification and Continued Airworthiness	21		
Holryod: Historical Overview of Environment-Induced Cracking in 7xxx Aluminum Alloys	36		
Colvin: MELD Manufacturing History of Aerospace Aluminum SCC Development and Testing	63		
McAdam: DSTG Intergranular Corrosion of 7000 series Aluminium Alloys	78		
Hallam: DSTL UK MOD Response to EAC of 7000 Series Al Alloys	96		
Zwerink: Netherlands NLR Perspective EAC in Aluminium Alloy 7085-T7452	106		
Barrett: Airbus Briefing	118		
Carvalho da Cunha: Embraer Environmentally Assisted Cracking of High- Strength 7000 Series Aluminum Alloys	136		
Yates: Lockheed Martin Experiences and Efforts	143		
Davis: Arconic Briefing	154		
Whelchel: Constellium EAC/SCC of 7xxx Aluminum Alloys in High Humidity Environments	160		
Scheuring: Kaiser Aluminum Environmentally Assisted Cracking	178		
Lamb: Universal Alloy Corporation Briefing	184		
Burns: University of Virginia Problem Definition and Knowledge Gaps for 7xxx EAC	189		
Robson: The University of Manchester Understanding Performance of New Generation 7xxx Aluminium Alloys	289		
Holroyd: EIC Testing of Commercial AI-Zn-Mg-Cu Alloys	313		
Friedersdorf: Luna Labs Measurement of Environmentally Assisted Cracking in Atmospheric Tests	325		
Dante: Southwest Research Institute Briefing	336		
Rausch: The Ohio State University Environmental Effects 7xxx Series Aluminum Alloys	346		
Ashby: CMI Beasy Software Tool Development and Capability Overview	358		



**U.S. AIR FORCE** 





## AFRL Perspective and Experience with Unexpected Environmentally Assisted Cracking in 7XXX Alloys

Materials and Manufacturing Directorate

C. Hunter, A. Rosenberger, K. Wertz



- 1. How has your organization bounded the problem definition of unexpected cracking in 7XXX aluminum alloys in service? If possible, provide a brief explanation and examples in a pre-competitive format.
- 2. What key influencing factors are suspected for the damage mechanism(s) identified above (e.g. humidity, temperature, steady-state stress, other)?
- 3. What are the 3-5 most critical questions that you think need to be answered?
  - What are your hypotheses about the problem and potential solutions to that problem?
  - Does your organization have the resources to answer the 3-5 questions?
  - If so, what is the timeline to address the 3-5 questions?
  - Will this information be shared with the community? How would the community validate your work so it can be used to the greatest impact?
  - Do you foresee any significant challenges with translating research results into practical applications (e.g. predictive modeling or the establishment of threshold criteria)?
- 4. What suggestions does your organization have for a community-wide standardized test to address the cracking problem, and could it be used as part of the material qualification/certification process?
- 5. Is there a suggested path forward for new alloy development?
- 6. Do you have suggestions for preferred community-wide collaboration mechanisms to share research progress or engineering solutions?

### ...and here are our answers:



## Bounding the Issue

How has your organization bounded the problem definition of unexpected cracking in 7XXX aluminum alloys in service? If possible, provide a brief explanation and examples in a pre-competitive format.

- Unexpected intergranular cracking has been observed on several USAF platforms (7085-T7452 forgings, others)
- Occurred in 7XXX alloys explicitly identified in EASA SIB-2018-04. Comparative fractography exhibited features similar to those shown in EASA bulletin
- Cracked components were not exposed to atypical service environments
- Sustained stress thought to be well-characterized considered superposition of residual manufacturing, installation, and service stresses (\*)
  - ASK: Any known experiences where EAC observed below published SCC thresholds?
- Material selection assumptions have been called into question due to these and other reported findings across the community





## **Critical Questions**

What are the 3-5 most critical questions that you think need to be answered?

- What is the best method to manage the lives of high-strength aluminum alloy components in aerospace applications?
- Does an effective test for EAC sensitivity exist that provides for use of high-strength aluminum alloys? Or does the community need to develop it?
- Under what conditions do environmentally-assisted cracks arrest?

Does your organization have the resources to answer the 3-5 questions?

• No, but have initiated exploratory efforts (discussed on next slides).

Will this information be shared with the community? How would the community validate your work so it can be used to the greatest impact?

• Yes, plan to publically release all findings to the extent possible.



## Approach

Accelerated. Ex: ASTM G44/G47 Aggressive Saline Environment

Current Industry Standard Testing

"Whole Life": Combined Effect of Multiple Mechanisms Service environments typically less aggressive CI- concentrations. Local crack tip environment unknown. Actual Damage

Mechanisms

Crack Incubation Think: Corrosion Pitting

Crack Initiation Think: Large Pits to Crack

Small and Short Crack Growth Think: Crack Growth at Microstructure Length Scale

Long Crack Growth Think: Crack Growth as Bulk Material Property Each mechanism may have unique response to known variables.

#### **Known Influence Factors**

Microstructure Environment

Microstructure

Environment

Composition Microstructure

Environment

Microstructure Environment

Composition

Stress

Composition

Stress

Stress

Composition

Stress





## AFRL Supported Research on Unexpected EAC

#### **EXPLORATORY EFFORTS ON "MODEL" ALLOYS AND PRODUCT FORMS:**

- Explore long crack growth rates as a function of environmental conditions (temperature and humidity) comparing a "susceptible" alloy 7085-T7452 to "non-susceptible" 7050-T7451. Effort will be accompanied by fractography and full material characterization (e.g. bulk and grain boundary). Additionally explore crack pre-cursor differences via a series of electrochemical polarization studies.
- 2. Perform crack arrest study on above alloys to test the hypothesis that EAC initiated cracks will not grow when local mechanical driving forces fall below the threshold stress intensity.
- 3. Investigate shortcomings of ASTM G47/ASTM G44 for current issue.

#### **RESULTS TO BE SHARED PUBLICALLY.**

### These studies are funded; estimated completion date is Dec 2025.



## Proposed Community-Wide Collaboration

Do you foresee any significant challenges with translating research results into practical applications?

- Challenge: Arrive at community consensus for new test methodologies + publish industry standard
- Challenge: Identifying and understanding mechanisms sufficiently to allow for management of in-service components
- Challenge: Discern the limits of applicability for current standard test methods (i.e. when are the ASTM G47/G44 test methods appropriate?)

Proposed Collaboration Ideas

Suggest TIM become an annual event with potential virtual check-ins at six month intervals to share on-going results and insights.





### We didn't address all the questions...

- 1. How has your organization bounded the problem definition of unexpected cracking in 7XXX aluminum alloys in service? If possible, provide a brief explanation and examples in a pre-competitive format.
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- 6. Do you have suggestions for preferred community-wide collaboration mechanisms to share research progress or engineering solutions?

### ...still a lot of unknowns.

# Thank you!

## **AFRL Research on Unexpected EAC**

#### Thermodynamic (CALPHAD) Assessment "Sensitive" Alloys identified in EASA SIB 2018-04



#### "Proof-of-Concept" Stress Measurement Technique at CHESS Synchrotron

High-fidelity stress measurement of double blind joint connected via interference fit





#### 11

## AFRL Supported Research on Unexpected EAC

Explore long crack growth rates as a function of environmental conditions (temperature and humidity)











AFRL Supported Research on Unexpected EAC

Dr. Henry Holroyd

AFRL

D 100 µm







Explore efficacy of stress intensity threshold values.

"Susceptible" 7085-T7452 "Non-susceptible" 7050-T7451

40°C	DRY	85	Primary Creep Exhaust (50/70%)
70°C	DRY	85	Primary Creep Exhaust (50/70%)
70°C	50	85	Crack Arrest



Federal Aviation Administration

# FAA Opening Remarks

**Presented at:** AFRL-FAA TIM on EAC / SCC of High Strength Al Alloys November 5, 2024 Dayton, OH

**Presented by:** 

Michael Gorelik, PhD

Chief Scientist for Fatigue and Damage Tolerance Federal Aviation Administration

# **Problem Statement**

- Premature cracking of certain high-strength 7000-series Al alloys used in aircraft structures
  - Lacking apparent correlation to time in-service (FC / FH)
  - Demonstrates attributes of EAC / SCC
  - Potential contributing factors: alloy chemistry, humidity, temperature, stress
    - Stress several interpretations → mission profile: cyclic vs. static; residual stress – mfg process; assembly stress – in-spec vs. out of spec
- Three areas of consideration:
  - Management of current fleet / COS
  - On-going production of previously certified aircraft models
  - Future TC / PC material selection / screening / Qual&Cert
    - TC Type Certificate; PC Production Certificate (FAA)
- Products affected (*confirmed*) Part 25 (multiple OEMs); Part 29 (select OEMs)



# **Problem Statement (cont.)**

- Potential elements of path forward:
  - R&D to address knowledge gaps (including potential coordinated multi-agency projects)
  - Consortium(s) or standards committee(s) to share "lessons learned" and promote standardization
  - Agency-specific actions (e.g. policy statements, guidance materials); regulatory harmonization
  - More formal regulatory vehicles ARC or ARAC (need TBD) ARC – Aviation Rulemaking Committee ARAC – Aviation Rulemaking Advisory Committee



## Sample Questions to be Addressed (not necessarily all during TIM)

- Are we dealing with single or multiple mechanisms?
- What is the range of affected Al alloys?
- What material attributes make them susceptible?
- Do we understand the *physics* of degradation mechanisms?
  - List of key contributing factors + rank-order
  - Can the onset and propagation of damage be *predicted or modeled*?
    - If yes what are the gaps? If not are "pass / fail" criteria the only option?
- Can we develop robust Qual&Cert test methods and acceptance criteria that will safeguard against the degradation mechanisms in question?
- Is it feasible to eliminate the use of EAC-susceptible alloys going forward?



# **Specific Expectations from TIM**

- Identify community of practice and establish dialogue Day 1
- Share technical information and "lessons learned" Day 1
  - Understanding of pre-competitive "threshold" for information sharing across the industry
- Initial summary of knowledge gaps Days 1, 2
- Initial definition of supporting / enabling R&D projects Day 2
  - e.g. pipeline of project topics, prioritization, collaborative R&D opportunities
- Discussion of viable collaborative mechanisms going forward – Day 2
  - Government industry academia
  - Civil vs. military aviation
  - Part 25 only, or broader?
- Interactive discussion re. the Sample Questions on p. 4 via three parallel breakout sessions – *Day 2*



# **Discussion**



Michael Gorelik, Ph.D., PMP

Federal Aviation Administration Chief Scientist, *Fatigue and Damage Tolerance* <u>michael.gorelik@faa.gov</u>







### **Certification & Continued Airworthiness**

AFRL FAA Technical Interchange Meeting (TIM) on Environmentally Assisted Cracking (EAC) / Stress Corrosion Cracking (SCC) of High Strength 7000 Series Aluminum Alloys,

5 6 Nov 2024, Dayton OH

Simon Waite Senior Expert – Materials EASA

Laurent Pinsard Chief Expert - Airframe EASA

05.11.2024

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## Content

- EAC history
- Impact on certification
- EASA Corrective Action (SIB)
- Impact on Continued Airworthiness
- Conclusion & improvement for future certification
- Standard to be adapted



EAC test

Figure 5 - Typical EAC fracture surface

**EASA** 



These cracks typically start from holes or other areas of stress concentration and usually propagate in a plane perpendicular to the short transverse (ST) direction. This phenomenon has been linked to the chemical composition of the alloy, notably a high zinc/magnesium ratio, combined with low copper content. Brittle fractures have been reproduced under laboratory environment and cracking has proven to be driven by time exposure (ageing) and is not fatigue related, although further crack propagation under operative loads cannot be excluded.

SCC test (ASTM G 47)











HYDROGEN ENVIRONMENT ASSISTED CRACKING OF AN AL-ZN-MG-(CU) ALLOY George A. Young Jr. August 1999



A possible scenario for a link between environment and hydrogen embrittlement (without corrosion products)





#### Safety Information Bulletin 2018-04

- 6 Aluminium Alloys are listed in EASA SIB : 7037, 7040\*, 7055, 7085, 7099, and 7449
- EAC phenomenon can occur only when the three following conditions are present: (1) susceptible material alloy, (2) sustained stress in the ST direction and (3) ageing in a typical environment
- EASA recommends all affected organisations to evaluate the extent of the issue, particularly to:
  - Identify components made of EAC sensitive aluminium alloys.
  - Evaluate the sensitivity to and criticality of EAC in the component.
  - Report these evaluation results to EASA.

MEADA

#### Note: SIB Revision 2018 04R2 March 2021:

- added 7140 (T7651 temper)
- provides a 'generic' test method that can be used to support identification of material susceptibility to this form of EAC.

## **H-EAC SIB**

Appendix 1 – EAC Generic Test Method

1. EAC generic test method and test conditions:

As suggested by public literature or published test methods:

#### 'Generic' test method could benefit from common protocol to optimise identifying critical temperature, environment, load etc for any particular application and alloy

#### Specimen:

- $\circ~$  Specimen geometry: Round smooth bar  $\rightarrow$  according to ASTM G49 and ASTM E8 Tension test
- o Align longitudinal axis of specimen (loading direction) with the Material ST grain direction

#### Loading:

- Loading device: Test rig → In Style of ASTM G49
- Loading: Constant load / displacement → according to ASTM G49
- Load level: set at 85% Yield only → In the style ASTM G64

#### **Environment:**

- Level of Humidity:  $85\% \pm 5\% \rightarrow \text{Ref. 1}$ )
- Temperature:  $70^{\circ}C \pm 1^{\circ}C \rightarrow Ref. 1$ )

#### Test time:

Test duration is recommended to be at least 100 days

Example: Adapt SCC test in accordance with ASTM G 47 Experimental Conditions •Specimen (typically): 60mm long round bar, dia. ~3mm •Load: Constant 25-75 % Rp0.2 •Environment: 3,5% NaCl alternate immersion •Temperature: RT



# **H-EAC Experience & findings**

- The intergranular fracture generated by H-EAC does not show any microscopic corrosion indication vs Stress Corrosion Cracking (pitting visible in the grains)
- Interference fit (stress) and environmental conditions (moisture and temperature) are contributing factors to HEAC - not easily monitored or quantified.
- Crack initiation and propagation does not follow conventional laws currently used in F&DT evaluation.
- H-EAC characteristics are unpredictability & variability. Even if the parameters are now well identified, significant dispersion/scatter are observed in service: different parts installed on the same aircraft can be severely or barely affected.
- The "quality" or the detectability of the cracks is an additional issue to be considered for the H-EAC inspection: NDT requested



# **EAC findings**

- Al 7085, 7075, 7449
- Perpendicular short transverse cracking combined with Interference fit
- Multiple cracks but parts randomly affected

#### **Related ADs:**

2019-0074: EC225 LP Main Rotor – Rotating Swashplate Yokes – Inspection / Rework / Service Life Limit

2019-0181R1: A350 Fuselage – Forward and Aft Cargo Door Latch Fitting External Lugs – Inspection / Modification

2021-0192: EC135B Main Rotor – Upper and Lower H legs – Service Life Limit

2022-0019: A380 Wings – Front and Rear Spars – Inspection



## **H-EAC Future Developments**

- Alloy selection can be "controlled" (avoid EAC materials high Zn/Mg ratio, low Cu etc)
- Improve understanding of crack growth HEAC cracks are Mode II vs Mode I for conventional F&DT?
- Complex approaches like J-integral to capture the stability of the crack tip?
- Continue to inform industry of progress, identified product CAW actions in progress
- need to better standardise/improve test methods, e.g. to identify key parameter values:
  - Loads
  - Environment
  - Temperature





# **Thanks for your attention!**



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HEAC **Outdoor Exposure test** S, Smm FET



#### **H-EAC History** σ σ 0 Typical fatigue crack Typical HEAC crack Mode I: Mode II: Mode III: Mode II dominant Mode I dominant Opening In-plane shear Out-of-plane shear Driven by direct stress Driven by shear stress

For HEAC cracks, the location of initiation and growth results in cracks perpendicular to the normal fatigue crack direction and parallel to the primary bending stresses. For this reason, Mode 2 becomes the dominant crack mechanism.



# **H-EAC Qualification**

SCC test in accordance with ASTM G 47

#### **Experimental Conditions**

- **Specimen** (typically): 60mm long round bar, dia. ~3mm
- Load: Constant 25-75 % Rp0.2
- Environment: 3,5% NaCl alternate immersion
- Temperature: RT

#### HEAC

Experimental conditions to be adapted

- Loads
- Environment
- Temperature



# Historical Overview of Environment-Induced Cracking of Al-Zn-Mg-Cu 7xxx series Aluminum Alloys

# Henry Holroyd

5<sup>th</sup> November 2024
# HH involvement with EIC of Aluminum Alloys

- 45+ years experience in Industry and Academia
- **1979** First publication on AI-Zn-Mg-Cu EIC:
- "Reduced Ductility of a High Strength Aluminium Alloy During and After Exposure to Water"

For next 42 years we believed this an early report of pre-exposure embrittlement for a commercial AI-Zn-Mg-Cu alloy

> In 2020 I became aware of a 1913 study published in German.

Several other unexpected findings during my deep-dive into 'History of EIC in aluminum alloys' (Covid-isolation in Southern Missouri in 2020/2021) EIC in Commercial Aluminum Alloys started in 1899 (~125 years ago, 45 years earlier than previously thought)

Reasons why pre-1944 EIC incidents not previously captured are:

**Alternate Descriptors:** 

- Spontaneous Disintegration
- Season Cracking of Aluminum
- Spannungskorrosion
- > Intergranular fracture under prolonged application of Stress
- Acceleration of corrosion under stress
- Stress Cracking and

Failure to appreciate work published in German, French, Japanese ...

# 1913 – Earliest EIC in Commercial Al-Zn-Mg-Cu Alloy during Structural Use

Al-9.4Zn-0.39Mg-0.32Cu (Zinc-Duralumin)

• Overhead Power Lines used Ore Mountain region of Germany

• Extrusions in German Zeppelin Airships bombing London in WWI

(Japanese Naval Admiral 'purloined' sample from a Zeppelin downed by British in Croydon was sent to Japan via 'Diplomatic Bag') Pre-exposure to water at 70°C and subsequent mechanical properties for Al-9.4Zn-0.39Mg-0.32Cu alloy tensile tested in air or water at RT and 70°C (1913)



Holroyd et al, Corrosion (2023)

# London WW1 – Zeppelin Frame 1916



# Early Al-Zn-Mg-Cu Alloy Development

- UK (NPL) 1919 initially active but later focus on other aluminum alloys
- Experimental alloys in US in late 1920's, efforts intensified during 1930's, stimulated by Military demand and UK's development of DTD 363 (Hiduminium RR77) at High-Duty Alloys in 1937
- Japanese studies lead to 'Extra Special Duralumin' (1936) used in Zero Fighter Planes (1938), subsequently exploited by US (1942) as basis for 75S, the precursor of AA7075

REVIEW

# History of the Development of Extra Super Duralumin and Future Research Issues of Al–Zn–Mg Alloys

#### Hideo Yoshida\*

#### ESD Laboratory, Nagoya 466-0005, Japan

Almost 85 years have passed since the development of Al–Zn–Mg–Cu Extra Super Duralumin in Japan in 1936. This alloy was developed by Dr. Igarashi in response to the Japanese Navy's order to Sumitomo to develop an alloy with tensile strength of 588 MPa (60 kg/mm<sup>2</sup>) or higher, exceeding Alcoa's super duralumin 24S. This alloy was then adopted for use in the Zero fighter Based on this alloy, Alcoa's 75S was developed in 1943 This paper describes the history of the development of Extra Super Duralumin, starting with Duralumin. The paper also discusses the issues that are considered important for the future development of the aluminum alloys based on the historical review and our study. One is the relationship between quench sensitivity and age-hardening properties of Al–Zn–Mg alloys and the second is the occurrence of shear fracture in the Al–Zn–Mg–(Cu)–Zr alloys. This is the points to be noted in the development of high strength and high toughness Al–Zn– Mg–Cu alloys. [doi:10.2320/matertrans.MT-LA2022019]

(Received May 30, 2022; Accepted July 21, 2022; Published January 25, 2023)

Keywords: Duralumin, Extra Super Duralumin, 2024, 7075, stress corrosion cracking, quench sensitivity, shear fracture



Effect of Cr addition on season cracking (SCC) of Al–Zn–Mg–(Cu) alloys. I. Igarashi: Doctoral Thesis, Osaka Imperial University, (1939)

# <u>1930's to mid-1940's</u>

#### Al-Zn-Mg-Cu Alloy development ongoing independently in US, UK, Germany and Japan

Alloy #	Year	Zn	Mg	Cu	Mn	Cr	UTS	YS	%	Envisaged	
							(MPa)	(MPa)	Elong	Product*	
X70S (US)	1931	10.0	0.4	1.0	0.7		400	310	18	F	
X71S US)	1932	10.0	2.0	2.0	1.0		585	560	10	S <i>,</i> E	
XB71S (US)	1932	9.0	2.7	2.0	1.0		595	565	10	S	
X73S (US)	1935	5.2	0.9	0.5			370	290	20	F	
DTD 363A (UK)	1937	5.5	2.8	1.3	0.7	0.5	586	510	5	F <i>,</i> E	
ESD (Japan)	1937	8.0	1.5	2.0	0.5	0.2	585		15	E, S	
ESD (Japan)	1020	6 9	1.2-	1.5-	0.3-	0.1-				ES	
	1939	0-0	1.8	2.5	1	0.4				E, 3	
X7C70S (US)	1938	8.0	1.1	0.5	0.8		480	420	17	F	
X74S (US)	1939	5.2	2.1	1.5	0.4		510	440	12	S	
FLW 3415	1042	4.5-	2.0-	0 5	0.2-	0.2	151	254	20	S E 14/	
(Germany)	1942	5.5	2.8	0.5	0.3	0.2	451	554	5.0	3, E, VV	
DTD 683 (RR77)			5 2-	2.2-	03-	0 18-					
(IIK)	1943	UK	5.2 6.2	2.2	0.5	0.10	541	463	7	S, F, E	
			0.2	5.20	0.7	0.7					
75S (US)	1943	5.6	2.5	1.6		0.25	570	500	11	S, P, F, E	

\*F = Forging, S = Sheet,

#### (1942- US access to Japanese ESD alloy from WW2 Zero Fighter Aircraft)

P = Plate, E = Extrusion



Alloy #	Year		Zn	Mg	Cu	Mn	Cr	other	UTS	YS	%	Product*
											Elong	
AA7178	1951	USA	6.3-	2.4-	1.6-	0.3	0.18-		600	540	10	S, P, E
			7.3	3.1	2.4		0.28					
AA7075	1954	USA	5.1-	2.1-	1.2-	0.3	0.18-		570	500	11	S, P, F, E
			6.1	2.9	2.0		0.4					
AA7079	<b>1954</b> §	USA	3.8-	2.9 -	0.4	0.1-	0.10-		540	470	14	S, P, F, E
			4.8	3.7	-0.8	0.3	0.25					
AA7001	1955	USA	6.8-	2.6-	1.6-	0.2	0.18-		675	625	9	E
			8.0	3.4	2.6		0.35					
AZ74	1958	Germany	5.5-	2.1-	0.7-	<0.1	0.15-	0.3-	590	539	9.8	F, P, E
			6.5	2.5	1.0		0.25	0.5 Ag				
X7080-T7	1965	USA	5.0-	1.5-	0.5-	0.1-	0.25		448	393	6	F
			7.0	3.0	1.5	0.7						
FLW 3415			4.5-	2.0-		0.2-						
	1942	Germany	55	28	0.5	03	0.2		451	354	3.8	S, E, W
			4.0	2.0		0.2	0.15					
FLVV 3423	1942	Germany	4.0-	3-		0.2-	0.15-					E
		/	4.5	3.5		0.6	0.25					

# <u>AA7075-T6 ---- AA7075-T73 ----AA7079-T6</u>

- By 1950, 75S (AA7075-T6) was 20% of Alcoa's total shipment of heat-treatable aircraft products.
- EIC in thicker material (ST loading direction) forced increased use of overaged duplex –T73 temper along with its associated ~15 % strength loss.
- In 1954 Alcoa introduced a 'promising' New Alloy AA7079-T6, believed then to provide:
- Higher short-transverse ductility and mechanical strength properties in thicksections than any other commercial aluminum alloy
- Improve EIC resistance relative to AA7075-T6, with cracking rarely initiating at stresses below 138 MPa during alternate immersion testing.
- AA7079-T6 usage grew rapidly during late 1950's to become in early 1960's the most used alloy for large highstrength forgings in the US
- EIC service issues during late 1950's, escalated to unprecedented levels during the 1960's

#### AA7079-T6 suffers EIC at ~ 50 MPa in Water Vapour (Duplex aging -T73 or RRA ineffective)



# Outfall of AA7079-T6 EIC Service Issues during the 1960's and early 1970's

<u>Unprecedented</u> R&D Funding from US Government Agencies flowed during the mid-1960's, through the 1970's into the early 1980's

Aspirational R&D Targets to address EIC performance included:

> Full characterization of the cracking phenomena

Provision of reliable accelerated EIC test methods

Establishment of alloy chemical compositions and manufacturing process routes to provide adequate EIC resistance under expected service conditions

# US Government Agency R&D Funding (1963-1982) to characterize EIC/SCC of Aluminum alloys

Date	Tittle	Funding Agency	Recipient	Reports
May 1963 – May 1966	Investigations of the Stress Corrosion Cracking of High Strength Aluminum Alloys	NASA	Alcoa	Sprowls et al, 1966 [85]
Dec 1963 – Feb. 1966	Investigations of the Mechanisms of Stress Corrosion of Aluminum Alloys	Bureau of Naval Weapons	Alcoa	G C English 1965 [83] J McHardy 1966 [84]
1965 – 1966	A Fundamental Investigation of the Nature of Stress- Corrosion Cracking of Aluminum Alloys	American Air Force	Battelle Memorial Institute	F H Haynie et al 1967 [86]
1966 – 1968	Studies of Crack Initiation Phenomena associated with Stress Corrosion of Aluminum Alloys	NASA	Alcoa	M S Hunter et al 1966 [87] M S Hunter & W G Frickle 1969 [88]
March 1966 – March 1969	Development of a Rapid Stress-Corrosion Test for Aluminum Alloys	NASA	Kaiser Aluminum & Chemical Corporation	W J Helfrich 1968 [93]
June 1966 – June 1969	Studies of the General Mechanism of the Stress Corrosion of Aluminum Alloys and Development of Techniques for its detection	NASA	Tyco Laboratories, Inc	S B Brummer et al [91]
1966	Development of Higher Strength Aluminum Alloys with Improved Stress Corrosion Resistance	Air Force Materials Laboratory	The Boeing Company	J C McMillan, M V Hyatt [89]
May 1967 – May 1968	The Role of Dislocations in the Stress-Corrosion Cracking of Aluminum Alloys	Naval Air System Command	Rocketdyne, Rockwell Corporation	A J Jacobs 1968 [90]
1967 – 1969	Investigation to Improve the Stress-Corrosion Resistance of Aluminum Alloys through Alloy Additions and Specialized Heat Treatment	Naval Air System Command	Alcoa	J T Staley 1969 [92]
May 1967 – March 1970	Development of a High- Strength, Stress Corrosion Resistant Aluminum Alloy for use in Thick Sections	Air Force Materials Laboratory	The Boeing Company	J C McMillan, M V Hyatt, 1967 [88], 1968 [99] M V Hyatt, H W Schimmelbusch, 1970 [100]
July 1968- Aug. 1973	Evaluation of Stress- Corrosion Cracking Susceptibility using Fracture Mechanics Techniques	NASA	Alcoa	D O Sprowls et al 1973 [94]
July 1969 -Feb. 1972	Investigation of Smooth Specimen SCC Test Procedure. Variation in Environment, Specimen size, Stressing Frame, and Stress State.	NASA	Alcoa	B F Lifka, D O Sprowls, R A Kelsey [95]
May 1967 – March 1970	Development of a High- Strength, Stress Corrosion Resistant Aluminum Alloy for use in Thick Sections	Air Force Materials Laboratory	The Boeing Company	M V Hyatt, H W Schimmelbusch, 1970 [100]
July 1969 – July 1970	High Strength Aluminum Alloy Development	Air Force Materials Laboratory	Reynolds Metal Co.	D S Thompson, S A Levy, 1970 [101]
1		1	1	1

1970	Exploratory Development of High-Strength, Stress Corrosion Resistant Aluminum Alloy for use in	Air Force Materials Laboratory	Alcoa	J T Staley, H Y Hunsicker [107]
1970	Thick Section Applications Investigation to Develop a High-Strength Stress- Corrosion Resistant Aluminum Aircraft Alloy	Naval Air Systems Command	Alcoa	J T Staley [106]
1970	Investigation to Develop a High-Strength Stress- Corrosion Resistant Aircraft Alloy	Naval Air Systems Command	Alcoa	J T Staley [90EE]
Sept. 1970 – Feb. 1972	Further Development of Aluminum Alloy X7050	Naval Air Systems Command	Alcoa	J T Staley, J P Lyle, H Y Hunsicker, 1972 [105]
June 1971 – Dec. 1972	Comparison of Aluminum Alloy 7050, 7049, MA52, and 7175.T736 Die Forgings	Air Force Materials Laboratory	Alcoa	J T Staley, 1972 [9102]
May 1972 – Nov. 1974	Design Mechanical Properties, Fracture Toughness, Fatigue Properties, Exfoliation and Stress Corrosion of 7050 Sheet, Plate, Hand Forgings, Dia Forgings and Extrusions	Naval Air Command	Alcoa	RE Davies, G E Nordmark, J D Walsh, 1975 [103]
March 1973 – June 1976	Aluminum Alloy 7050 Extrusions	Air Force Materials Laboratory	Alcoa	J T Staley et al, 1977 [104]
1980	Seacoast Stress Corrosion Cracking of Aluminum Alloys	NASA	NASA	TS Humphries, E E Nelson, 1981 [196]
March 1981 — May 1982	A Study of Environmental Characterization of Conventional and Advanced Aluminum Alloys for Selection and Design Part 1: Literature Review Part 2: The Breaking-Load Test Method	NASA	Alcoa	D O Sprowls, 1984 [97] D O Sprowls et al, 1984 [98]

# How successful was the R&D Investment?

- Evolution and emergence of AA7050 and its multi-decade essentially EICfree service performance is a major positive
- Progress on the EIC aspirational targets less impressive:
- Full characterization of the phenomena (Still only partially achieved)
- To provide reliable accelerated EIC test methods (Still awaiting major progress)
- Establishment of alloy chemical compositions and manufacturing process routes to provide adequate (Al EIC resistance under expected service conditions

(Alloy Development now limited by Zinc content Issue)

# **Current Standard EIC Test Methods**

#### 2017 Russ Jones

ASM Book Stress Corrosion Cracking–Material Performance and Evaluation, 2<sup>nd</sup> Ed. Chapter 17: Evaluation of Stress Corrosion Cracking

"One of the toughest problems for SCC investigators is that of convincing the decision makers that service life cannot be predicted in hard numbers because material traditionally have been evaluated by comparison"

#### HH et al (2024)

ASTM G47, G103, G129, G139 and G168 are unable to reliable differentiate between the EIC susceptibility of relatively resistant Al-Zn-Mg-Cu aluminum alloys.

# Why are Today's EIC Tests Methods Inadequate?

**EIC** performances evaluated on the basis of comparison

> Environmental test conditions not relatable to expected 'Service Conditions'

EIC initiation/early-stage growth rarely accessed, despite controlling EIC performance in structural applications

> Initial surface conditions non-relatable or representative of expected service use

> Complacency following AA7050 having no significant EIC Service Issues over decades

> Low-copper content Al-Zn-Mg-Cu alloys (AA7079, AA7039, AA7020) not the only a 'Special Case'

Failure to react to experimental evidence (e.g. Staley, 1973) Al-Zn-Mg-Cu alloys with higher-zinc contents than AA7050 have EIC propensities in natural atmospheres' undetected during standard ASTM G47 testing in alternate immersion saline solutions.

ASTM G47, G103, G129, G139 and G168 testing fail to reliable differentiate between EIC susceptibility of relatively resistant Al-Zn-Mg-Cu aluminum alloys tempers

#### Experienced–Based EIC Rating for Aluminum Alloys suggested by Sprowls et al (1973) using Smooth and Pre-Cracked Test Specimen Data

EIC Rating Susceptibility	EIC Threshold		EIC K-Insensitive Growth Bate (m/s)	Exfoliation Rating (ASTM G34)
	б <sub>ыс</sub> (MPa) % Yield Stress (ASTM G47)	K <sub>IEIC</sub> % K <sub>IC</sub> (DCB)		
A– Very Low	>90	>95	< 7 x 10 <sup>-11</sup>	Р
B– Low	>75	>80	7 x 10 <sup>-11</sup> to 3 x 10 <sup>-10</sup>	P/EA
C–Moderate	>40	>50	3 x 10 <sup>-10</sup> to 3 x 10 <sup>-9</sup>	EA/EB
D-Appreciable	<40	<50	>3 x 10 <sup>-9</sup>	EB/EC

### Experienced–Based EIC Rating for Aluminum Alloys suggested by Sprowls, et al (1973) using Smooth and Pre-Cracked Test Specimen Data

EIC Rating	EIC Thre	shold	EIC	Exfoliation	
Susceptibility			K-Insensitive	Rating	
			Growth Rate	(ASTM G34)	
	б <sub>ыс</sub> (МРа)	KIEIC	(m/s)		i ypical Alloy
	% Yield Stress	% K <sub>IC</sub>			
	(ASTM G47)	(DCB)			
A– Very Low	>90	>95	< 7 x 10 <sup>-11</sup>	Р	6061-T6
B– Low	>75	>80	7 x 10 <sup>-11</sup> to		7075-T73,
			3 x 10 <sup>-10</sup>	P/CA	7050-T73
C–Moderate	>40	>50	3 x 10 <sup>-10</sup> to		7075-T76,
			3 x 10 <sup>-9</sup>	EA/ED	7050-T76
D-Appreciable	<40	<50	>3 x 10 <sup>-9</sup>		7055-T77,
					7085-T76

## Next Generation EIC Test Methods?

• Tests should be relatable to expected service conditions and focus on crack 'initiation', early-stage growth and crack 'arrest' behavior.



Holroyd et al., Corrosion (2023)



#### Holroyd et al., Corrosion (2023)

# Al-Zn-Mg-Cu alloy Development Next Generation?

• Optimize current 'Latest Generation' Alloys first?

• Biggest Challenge is overcoming Maximum Zinc Content issue

## Next generation Al-Zn-Mg-Cu Alloys will Require Understanding of Zinc Content Ceiling Issue



\*Over-aging heat-treatments to reduce EIC becoming less effective with increasing Total Zinc content and non-existent for Zn > ~9 wt%



Holroyd et al., Corrosion (2023)

# History of Environment-Induced Cracking of Al-Zn-Mg-Cu 7xxx series Aluminum Alloys

<u>Review Paper Published in January Issue of Corrosion (2023)</u>

**Environment-Induced Cracking of High-Strength Al-Zn-Mg-Cu** Aluminum Alloys: Past, Present, and Future

(Henry Holroyd, Tim Burnett, John Lewandowski and Geoff Scamans)

(Is a 1960's type EIC experience re-emerging?)

# History of Aerospace Aluminum SCC Development and Testing

November 5, 2024

**Ed Colvin** 

Chief Technical Officer - MELD Manufacturing President & Consultant - HELM Technology, LLC Retired VP Technology – Arconic/Howmet Aerospace

## **Aluminum Corrosion and EAC**

There is no aluminum metal in nature, so eventually all the aluminum we have will corrode.

- Inevitability The 2<sup>nd</sup> law of thermodynamics tells us that all aluminum metal will oxidize
- Localized Corrosion Microstructural-mechanical-environmental interactions determine what part will corrode first and the resulting morphology
- Timing Reaction kinetics, protection systems, and electrochemical factors tell us how fast
- Consequences Structural design, service requirements, design life, etc. determine what matters.

In other words, it's all going to corrode, it's just a question of which part corrodes first, how fast, and what are the consequences?





#### **Great People I've Been Fortunate Enough to Know Personally**

During roles that included Corrosion Characterization, Corrosion & EAC Test Development, Alloy Development, Ingot Casting, Rolling, Extrusion & Forging Technology:

Don Sprowls	Paul Ziman
Mike Hyatt	John Liu
Markus Speidel	Bob Bucci
Tom Summerson	Glenn Stoner
Bernie Lifka	Ed Starke
Basil Ponchel	Howard Pickering
Henry Holroyd	plus many, many others
Jack Snodgrass	
Jim Staley	

## **Two Key ASTM Special Technical Publications**

STRESS CORROSION-		
NEW APPROACHES		
	NEW METH	ODS FOR
	CORRO	SION
	TESTI	VG OF
H. L. CRAIG, Jr., editor	ALUM	NUM
	ALLO	)YS
	AGARWALA / G	JGIANSKY
STP 610		
ditt.	STP 113	4
ALL AMERICAN SOCIETY FOR TESTING AND MATERIALS	451	
Symposium	Sympo	osium
June 22-27, 1975	May 21-2	22, 1990
Montreal, Canada	San Fran	cisco, CA

## 2x24 & 7x75 Alloys – Thin Products that Became Thick

Table 3.2.3.0(a). Mate	erial Specifications for 2024 Aluminum	Specification	Form
Alloy Specification	Form	AMS 4044 AMS 4045 AMS 4078 AMS-OO-A-250/12, 24	Bare sheet and plate Bare sheet and plate Bare plate Bare sheet and plate
AMS 4037	Bare sheet and plate	AMS-QQ-A-250/13, 25	Clad sheet and plate
AMS 4035	Bare sheet and plate	AMS 4049	Clad sheet and plate
AMS-QQ-A-250/4	Bare sheet and plate	AMS 4122	Bar and rod, rolled or cold finished
AMS-QQ-A-250/5	Clad sheet and plate	AMS 4123 AMS 4124	Bar and rod, rolled or cold finished Bar and rod, rolled or cold finished
AMS 4120	Bar and rod, rolled or cold-finished	AMS 4186	Bar and rod, rolled or cold finished
AMS-QQ-A-225/6	Rolled or drawn bar, rod, and wire	AMS 4187	Bar and rod, rolled or cold finished
AMS 4086	Tubing, hydraulic, seamless, drawn	AMS-QQ-A-225/9	Rolled or drawn bar and rod
AMS-WW-T-700/3	Tubing	AMS-QQ-A-200/11, 15	Extruded bar, rod, and shapes
AMS 4152	Extrusion	AIMS 4120	Forging
AMS 4164	Extrusion	AMS 4141	Die forging
AMS 4165	Extrusion	AMS 4147	Forging
AMS-QQ-A-200/3	Extruded bar, rod, and shapes	AMS-A-22771 AMS-00-A-367	Forging

Table 3.7.6.0(a). Material Specifications for 7075 Aluminum Alloy

Both alloys were developed for thin sections in tempers intended for sheet and thin extrusions, 2024-T3X and 7075-T6X, which kept stresses in the L and LT directions, but still SCC was a key factor during development.

Aircraft developed in the 1950s required heavy section products making it possible to stress the ST direction and necessitating new test procedures.

### **Requirements for Large Scale Test Procedure**

Capability to test thousands of specimens simultaneously.

Similar specimens and stress procedure to be used in natural and service environments

Test all grain orientations

One environment for as many alloy-temper combinations as possible

Objective failure criteria that can be used for development, qualification, quality assurance, and surveillance testing

Not intended to address specific crack growth mechanisms.

*Non-requirement – perfect correlation with any specific service environment as there are an infinite number of service environments.* 



## ASTM G47 – Alternate Immersion in 3.5% NaCl

#### Standard Test Method for Determining Susceptibility to Stress-Corrosion Cracking of 2XXX and 7XXX Aluminum Alloy Products<sup>1</sup>

This standard is issued under the fixed designation G47; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

#### Cyclic exposure to 3.5% NaCl in a controlled humidity to produce wet-dry cycles that mimic natural and service environments.

Small specimens emphasize crack initiation rather than crack growth.

#### Daily inspection for cracking.

Differentiation between cracking and IG corrosion.

Alternative solutions such as artificial seawater.

#### 4. Significance and Use

4.1 The 3.5 % NaCl solution alternate immersion test provides a test environment for detecting materials that would be likely to be susceptible to SCC in natural outdoor environments, especially environments with marine influences.<sup>3,4,5</sup> For determining actual serviceability of a material, other stress-corrosion tests should be performed in the intended service environment under conditions relating to the end use, including protective measures.

#### 13. Precision and Bias

13.2 *Bias*—The procedure in Test Method G47 has no bias because the result of the pass-fail stress-corrosion cracking test is defined only in terms of this test method.

## **Compromises Required for a Single Environment**

## G47 was intended for comparative rankings in a single environment that correlated with the seacoast.

- From the start they knew that 2xxx & 7xxx alloy relative life expectancies were not the same AI vs. seacoast.
- Industrial and acid environments produced different life expectancies.
- Alloy response within the 7xxx family varied, 7075 vs. 7079.

## Stressing techniques based on thickness and need for long-term outdoor exposure.

- Window frame
- C-rings
- Sheet bend specimens



## **Learnings and Developments from Alloy 7050**

The correlation between G47 and seacoast exposure was not the same for 2x24 and 7x75.

7050 showed a slightly different correlation than 7075.

Eventually 7050 will experience more failures severe seacoast locations than in G47.

Strength-conductivity relationship developed during the 7050 efforts



Short-Transverse Stress-Corrosion Tests of 7050-T7451X Extruded Shapes 1.2-2.93 in. (30-74 mm) Thick



Proposed Stress Corrosion Factor, SCF, (Sloping Line) Acceptance Criteria and Summary of Stress Corrosion Performance of 7050-T7451 Plate Tested at 35 ksi (241 MPa)

## **Resulting Ratings Based on G47 as Described in G64**

#### TABLE 1 Practical Interpretation of Ratings for Resistance to SCC

Note 1—The stress levels mentioned below and the test stresses mentioned in 6.2 are not to be interpreted as "threshold" stresses, and are not recommended for design. Other documents, such as MIL-HANDBOOK-5, MIL-STD-1568, NASC SD-24, and MSFC-SPEC-522A, should be consulted for design recommendations.

Rating	Interpretation
Α	Very high. SCC not anticipated in general applications if the total sustained tensile stress <sup>4</sup> is less than 75 % of the minimum specified yield strength for the alloy, heat treatment, product form, and orientation.
В	High. SCC not anticipated if the total sustained tensile stress <sup>A</sup> is less than 50 % of the minimum specified yield strength.
С	Intermediate. SCC not anticipated if the total sustained tensile stress <sup>A</sup> is less than 25 % of the minimum specified yield strength. This rating is designated for the short transverse direction in improved products used primarily for high resistance to exfoliation corrosion in relatively thin structures where appreciable short transverse stresses are unlikely.
D	Low. SCC failures have occurred in service or would be anticipated if there is any sustained tensile stress <sup>4</sup> in the designated test direction. This rating cur-

<sup>A</sup> The sum of all stresses including those from service loads (applied), heat treatment, straightening, forming, and so forth.

	Alloy and Temper <sup>4</sup>	Test Direction <sup>B</sup>	Rolled Plate	and Bar <sup>C</sup>	Extruded Shapes	Forgings
	7049-T76	L	D	D	A	D
Poviou of the rating tables immediately shows		LT	D	D	A	D
Review of the ruting tubles inineutitely shows		ST	D	D	С	D
the importance of not just alloy, but also	7149-T73	L	D	D	A	A
		LT	D	D	A	A
temper, product form, and stress direction.		ST	D	D	В	A
	7050-T74	L	А	D	A	A
		LT	А	D	А	A
		ST	В	D	В	B
	7050- <b>T</b> 76	L	А	A	A	D
		LT	A	В	A	D
		ST	C	В	C	D
	7075-T6	Ĺ	A	Ā	A	A
		LT	BE	D	BE	BE
		ST	D	D	D	D
	7075-T73	É.	A	A	A	A
		LT	A	A	A	A
		ST	A	A	A	A
# Rise of Precracked Specimens - 7075-T6X vs. 7079-T6X

7079-T6X showed more severe SCC in service than 7075-T6X, despite no more susceptibility in alternate immersion.

The seacoast did not differentiate the alloys properly either.

It took a long time, but eventually industrial environments, acid rain, showed a difference between the two alloys.

One key aspect was initiation time relative to crack growth rate.

DCB testing really shows the difference, 7079-T6X has much exhibits much faster crack growth rates in saline environments than 7075-T6X.

Notably the crack growth in saline environments is largely independent of exposure conditions.

G168 covers DCB testing.

Work by both Hyatt and later Lukasak, et al., shows that crack growth is a factor in smooth-specimen G47 time-tofailure, in fact, the Breaking Load method, G139, can show crack growth rates.



FIGURE 12 - K<sub>1</sub>-rate data for DCB specimens tested at two laboratories. The 7079-T651 specimens were from the same piece of 1-inch thick plate, whereas the 7075-T651 specimens were from 1-inch thick plate from different heats.

(4) Hyatt, M. V., "Use of Precracked Specimens in Stress-Corrosion Testing of High Strength Aluminum Alloys," *Corrosion*, Vol 26, No. 11, November 1970, pp. 487-503.

# **Barriers to Change**

There was a lot of corrosion test method development in the 1980s and 1990s, but minimal implementation.

Have to test thousands of specimens in numerous environments and compare to incumbent products.

Quality assurance tests have to be relatively quick and use limited material.

Existing specifications make any sort of change difficult even for new alloys.







FIG. 6 Examples of Exfoliation Rating ED (Very Severe) (Similar to EC Except for Much Greater Penetration and Loss



# **Current Role - MELD Manufacturing, Chief Technical Officer**







# **ELD** MANUFACTURING

Additive Friction Stir Deposition (AFSD) equipment, processes and components.

# AA 7075 Printing – One Reason I'm Still Interested in SCC





November 14, 2024

# Thank you very much!



Australian Government
Department of Defence
Defence Science and Technology Group

# DSTG Intergranular Corrosion of 7000 series aluminium alloys



#### Grant M<sup>c</sup>Adam, Alison Wythe, Chris Loader

Presented at the Technical Interchange Meeting on Environmentally Assisted Cracking of 7XXX Aluminum, Dayton, Ohio 5-6 Nov. 2024

Defending Australia and its National Interests www.defence.gov.au



# DSTG Program

- Outdoor exposure uncoated
- Outdoor exposure coated
- Thermal exposure
- Use of Corrosion Prevention Compounds (CPCs)
- In-service failures
- Laboratory grown intergranular corrosion (IGC)
- IGC and Fatigue

2

# **Outdoor Exposure - Uncoated**

#### **Research Summary**

- Range of Environments
- Different storage locations
- Different Alloys AA2024 AA2124 AA7050 AA7085



#### **Risks/Issues**

 Potential for corrosion not experienced on legacy alloys

#### DSTG Responsibilities

- Conduct Exposure
- Assess extent of Corrosion



#### Outcomes

- IGC greater than expected
- Shelter more severe than open
  - Wetter for longer and no removal of salt build-up by rain washing

#### **Outdoor Exposure – Uncoated - continued**



4

Open

SH = Shelter HA = Hangar

100

#### **Research Summary**

Assessing the ability of coatings systems to prevent corrosion

#### **Risks/Issues**

 Performance of chromate-free primer does not prevent corrosion

#### **DSTG Responsibilities**

 Outdoor performance testing of coated AA7085 against chromate containing controls



AA7085 Bare

AA2024 + Anodize + Non-Chormate Primer + Topcoat

AA7085 + Anodize + Non-Chromate Primer + Topcoat

AA2024 + Anodize + Chromated Primer + Topcoat

AA7085 + Anodize + Chromated Primer + Topcoat

#### Outcomes

• IGC continued to progress with time



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	3 months		6 months		12 months	
	Max. IG Length (µm)	Max. IG Depth (µm)	Max. IG Length (µm)	Max. IG Depth (µm)	Max. IG Length (µm)	Max. IG Depth (µm)
Open	229	120	416	96	420	113
Shelter	559	98	1271	172	1651	197
Hangar	31	20	-	-	71	48



#### **Risks/Issues Research Summary** Range of durations and temperatures assessed for Does repeated thermal cycling lead to increases in • impact on corrosion performance corrosion **DSTG Responsibilities** Outcomes Alloys: AA7050, AA7085 Thermal cycling does lead to increases in corrosion damage Thermal exposure: 1 & 10 h at 121 °C 450 AA7085 1 & 10 h at 177 °C 400 350 Corrosion Tests: Electrochemical <u>ال</u> 300 Susceptibility to exfoliation corrosion Depth 250 Droplet testing 200 200 150 150 100 50 AA7085 after 10 h at 177 °C 1 h, 121 °C 10 h, 121 °C 1 h, 177 °C **OFFICIAL** 10 h, 177 °C As received

Thermal Exposure

## **IGC - Summary**

#### **Research Summary**

- Aluminium Alloy 7085
  - Faster, cheaper forging
  - Susceptible to IGC
  - DSTG convinced Defence of need for corrosion mitigation strategy
  - Long term outdoor exposure of AA7085 with coating stack-up to evaluate performance

#### **DSTG Responsibilities**

- Outdoor exposure testing of AA7085
- Conduct trial to assess the effectiveness of NDI techniques and corrosion repairs to remove IGC

# 50 μm

#### **Risks/Issues**

- NDI techniques do not reliably detect IGC
- Repairs of corrosion do not effectively remove IGC damage and it is the initiation point for future corrosion

#### Outcomes

- Improved understanding of the impact of IGC on maintenance activities
- Advice on risk of continued operations in the presence of IGC
- Advice on repair procedure ability to manage corrosion in bulkhead material
- Assessment of unserviceable component due to corrosion damage (AA 7050)

#### **Research Summary**

- Fastener hole corrosion treated with CPC to arrest corrosion
- Are CPCs affective at arresting pitting and IGC

#### **Risks/Issues**

- CPCs are ineffective at arresting corrosion
- On-going corrosion related maintenance burden likely to increase

#### DSTG Responsibilities

- Assessed CPC (Esgard) that was being used by the RAAF
- Extend program to a CPC recommended by the Original Equipment Manufacturer



#### Outcomes

- Rapid assessment of CPCs for corrosion deferral
- Support introduction of CPCs as standard repairs within the program
- Use of CPCs will reduce the incidence and extent of fastener hole corrosion reducing the maintenance burden

#### **In-service corrosion example - 1**

- Significant attempts to remove surface corrosion from AA7050 had taken place before component was deemed unserviceable
- DSTG tasked to characterise remaining corrosion







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# **Findings Summary**

- Significant corrosion penetrations from hole bore
- Maximum penetration approx. 1200 μm
- Corrosion occurring in satellite holes
- Corrosion detected underneath coatings and sealants
- Presence of fine IGC networks confirmed, which are difficult to detect without destructive analysis
- Corrosion identified in a hole, not flagged as having corrosion present

#### **In-service corrosion example - 2**

- Cut outs from failed component provided to DSTG for destructive analysis
- Corrosion <u>had not</u> been cleared during the course of the maintenance on the aircraft
- Corrosion was still being detected
  - Never got to a clearing point within permitted damage limits and was the reason the material had to be excised
  - Bathtub repairs implemented
- Material AA7050



## **IGC Growth Experiments**

#### **Research Summary**

- To grow IGC down fastener holes on AA7085 that
  - a) Grows at a predictable rate
  - b) Represents characteristic <u>form</u> of fleet corrosion
  - c) Represents characteristic <u>size</u> of fleet corrosion

#### **DSTG Responsibilities**

- Grow IGC guided by in-service corrosion
- Adapt ASTM G110 or develop electrochemical methods to grow representative IGC
- Acknowledgement: Maria Salagaras, Nick Tugwell & Rainier Catubig

#### **Risks/Issues**

- Not possible to replicate microstructure in form and size
- IGC adversely impacts fatigue

#### Outcomes

Development of a test to representatively grow IGC



In-service corrosion on AA7050



IGC as found in-service



Extensive IGC or pitting

Lab-grown corrosion on AA7085 with ASTM G110 solution

## **IGC Summary**

- ASTM G110 Standard method for evaluating Intergranular corrosion resistance of 7000 series Aluminum alloys
- Immersion of AA7085 specimens in a solution containing NaCl and H<sub>2</sub>O<sub>2</sub> as described in ASTM G110 for times ranging 6 - 96 h
- Results so far;
  - 300 µm fine IGC networks after 24 h
  - Maximum penetration of 500 µm, after 96 h (4x exposure), with significantly less IGC present
- Beyond 24 h exposure time, penetration from hole slows down significantly, and tendency to form bulk pits increases
- Future work: refine procedure to grow larger, finer networks, rather than bulk pits

# Fatigue Crack Growth (FCG) from IGC and Corrosion

- Aim: better understand the risk posed by fatigue cracks growing from corrosion pits/IGC
- Method:
  - Introduce corrosion/IGC at hole in AA7085 coupons, then fatigue test
  - Perform QF to document fatigue nucleation and growth
  - Compare non-corroded to corroded
  - Acknowledgement: Isaac Field & Ben Dixon

#### **Test Matrix**

Orientation	Hole condition	Stress (MPa)	Coupons
L-S	As-machined	≈ 130	5
L-S	Corroded	≈ 130	5
T-L	As-machined	≈ 130	5
T-L	Corroded	≈ 130	5
S-L	As-machined	≈ 130	5
S-L	Corroded	≈ 130	5



Coupons ≈ 160 mm × 80 × mm x 5 mm 3 orthogonal orientations from 6" thick AA7085 forging



- DSTG Program
  - Outdoor exposure uncoated
  - Outdoor exposure coated
  - Thermal exposure
  - Use of CPCs
  - In-service failures
  - Laboratory grown IGC
  - IGC and Fatigue



- The authors would like to acknowledge the rest of our team whose research has contributed to this presentation:
  - Maria Salagaras
  - Nick Tugwell
  - Rainier Catubig
  - Ben Dixon
  - Isaac Field

# **Questions?**





# UK MOD Response to EAC of 7000 series Al Alloys

Dr David Hallam, Air Work Package Technical Lead, Dstl Advanced Materials Programme Prepared by: Joe Plummer, Dstl Senior Scientist



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**UK OFFICIAL** 

 1710 Naval Air Squadron and Dstl carried out rapid investigation of coupons from LII airframe forging (7085 T7452) based on EASA SIB 2014-04

- Testing was primarily to confirm susceptibility to cracking
- Results showed positive result for EAC in 7085 (T74)
- University of Manchester tasked through DMEx to deliver two year task on EAC for defence aviation-specific alloys and environments
  - Aims to understand effect of environmental factors (inc chloride exposure) to crack life cycle of 7085 T7452

The Science Inside

2

- Project will develop test procedures to allow real-time monitoring through life of crack (initiation, short to long cracks)
- Will also explore generation of statistical data sets to inform digital twin/active learning for crack initiation and growth – to support inspection regimes and potential crack management approaches
- UK MOD interested in engaging on international research collaboration
  - DMEx designed to facilitate IRC, including exchange of staff and collaborative projects UK OFFICIAL

#### Current UK MOD Involvement in EAC Issues

#### Lightning II Delivery Team – DE&S

- Providing representative material (forgings) for ongoing testing 1710 NAS & University of Manchester.
- Convened meeting with JPO on publication of EASA SIB
- Participation in F-35 Corrosion Prevention Advisory Board and Aircraft Structural Integrity Program meetings

#### 1710 Naval Air Squadron

- Carried out initial testing against EASA SIB
- Providing support to in-service platform failures and issues
- Participating in JSF Corrosion Prevention Advisory Board

#### Dstl

- Formulating and delivering S&T research related to EAC
- Supported initial testing against EASA SIB
- Engaging in international materials S&T collaboration

The Science Inside

- Once made aware of the SIB publication, 1710 Naval Air Squadron and Dstl carried out a rapid investigation of coupons from LII airframe forging (7085 T7452)
  - Testing was primarily to confirm susceptibility to cracking (with quick time to failure), not to develop understanding of initiation or growth of cracking
- C-rings (as specified in ASTM G38) produced to load in the ST grain direction
  - 6x c-rings produced per material
- Samples loaded and placed in conditioning chamber, with aim to run for 100 days
- Samples examined every 7 days to identify crack development

Materials	7085 T7452 7085 T7451 7050 T7451
Temperature	70°C+/-1°C
Humidity	85%+/-5%
Stress	85% YS

The Science Inside

Test conditions for UK MOD replication OFFICIAL of EASA SIB 2014-04

After 10 weeks, 4x7085 forgings and 3x7085 plates developed cracking

- Experiment halted at 12 weeks due to cabinet failure
- Micrographs revealed features aligned with brittle intergranular fracture as seen in the SIB



7085 T7452 forging fracture surface, including GB precipitates standing proud



7085 T7452 forging fracture surface, including gaping GBs

5 UK OFFICIAL

dstl The Science Inside

- MOD planned to repeat c-ring tests and run for full 100 days, however possibility of EAC in F-35 structure and output from University of Manchester viewed as sufficient evidence to proceed with research task
  - Initial Manchester work focused on tempers and conditions for civil aviation applications.
     Direct tasking required to address UK MOD key questions
- Initial research on susceptibility of alloys of interest to MOD planned between Dstl, 1710 and University of Manchester
  - Issues with recruitment prevented work from commencing
- New research task with Manchester now planned through the Dstl Defence Materials Centre of Excellence (DMEx)
  - Understand susceptibility of in-service alloys for defence aviation
  - Rebuild metallurgy expertise necessary to support development and selection of future alloys<sub>6</sub>

The Science Inside

# DAMEX Defence Materials Centre of Excellence





Dstl contract with the University of Manchester

UoM the legal entity that leads the Henry Royce Institute (HRI) - UK's national institute for advanced materials research and innovation

£42.5M over 5 years from 01/12/2023 (includes S&T, Man & Gov)

HRI a Hub and Spoke model – UoM the hub

Dstl's Centre is HRI plus:

- Other university centres of expertise in important fields, e.g. composites, high temperature testing...
- High Value Manufacturing Catapult
- Satellite Applications Catapult
- Research and Technology Organisations
- Industry

**UK OFFICIAL** 

#### A partnership

- University of Manchester tasked through DMEx to deliver two year project on EAC for defence platforms
  - Aims to understand effect of environmental factors (including chloride exposure) to crack life cycle of 7085 T7452
  - Project will develop test procedures to allow real-time monitoring through life of crack (initiation, short to long cracks)
  - Will also explore generation of statistical data sets to inform digital twin/active learning for crack initiation and growth – to support inspection regimes and potential crack management approaches
- UK MOD (Dstl, 1710 NAS and LII DT) to provide ongoing support to task and aid translation to in-service support
- Future work may support wider Air Materials efforts:
  - Potential to further develop models as part of Air Digital Twin thread
  - Developed tests and models could be utilised to de-risk development of future materials
    - Within scope of DMEx to support MOD "intelligent customer" status, but not lead development of new alloys

The Science Inside

• UK MOD interested in engaging on international research collaboration

- DMEx designed to facilitate IRC, including exchange of staff and collaborative projects
- Multiple routes to information sharing and indirect collaboration
  - Dstl utilise range of programmes and MOUs inc. TTCP, NATO AVT and AUKUS
  - 1710 active in JSF CPAB
- Direct collaboration (including sharing materials and data) in a timely manner will require planning and selection of suitable IRC programme
  - Recommend early engagement with relevant IRC leads to identify appropriate channel

The Science Inside



Discover more



10 UK OFFICIAL

# Dedicated to innovation in aerospace

(ntr

#### NLR perspective EAC in aluminium alloy 7085-T7452

Borit Zwerink 5 November 2024 Dayton, Ohio Dedicated to innovation in aerospace







3


#### Aerospace systems



Aerospace operations



#### Aerospace vehicles



Photo credits: ESA – P. Carril , 2012

Λ







**Predictive Mx** 



NDI/thermography



Mx tooling



**SHM/Impact Detection** 

6



**ECTM** 



**Data analytics** 



7

# Environmentally Assisted Cracking

- EAC according to European Union Aviation Safety Agency (EASA)
  - Issued from 2018 Safety Information Bulletin (SIB)

EASA SIB No.: 2018-04R2



Safety Information Bulletin

Airworthiness SIB No.: 2018-04R2 Issued: 04 March 2021

Subject:

Environmentally Assisted Cracking in certain Aluminium Alloys

Revision:

This SIB revises EASA SIB 2018-04R1 dated 13 September 2018.

Ref. Publications:

None.

#### Applicability:

Type Certificate (TC) holders, Supplemental Type Certificate holders, equipment manufacturers, maintenance organisations, production organisations and aluminium alloy producers.

#### Description:

EASA received reports of brittle cracking of aluminium alloy components. Additional investigation of some new generation 7xxx series alloys has shown that these have a sensitivity to a phenomenon known as environmentally assisted cracking (EAC), when subject to certain conditions in the normal operating environment. The type of EAC encountered appears to be caused by hydrogen embrittlement along the grain boundaries, leading to crack initiation and subsequent propagation. These cracks typically start from holes or other areas of stress concentration and usually propagate in a plane perpendicular to the short transverse (ST) direction. This phenomenon has been linked to the chemical composition of the alloy, notably a high zinc/magnesium ratio, combined with low copper content. Brittle fractures have been reproduced under laboratory environment and cracking has proven to be driven by time exposure (ageing) and is not fatigue related, although further crack propagation under operative loads cannot be excluded.

- Brittle cracking
- Certain conditions in the normal operating environment
- Propagate in ST direction
  - High zinc/magnesium ratio, combined with low copper content
- Not fatigue related
- No obvious corrosion reaction

# Testing method proposed in SIB

#### Specimen:

- $_{\odot}\,$  Specimen geometry: Round smooth bar ightarrow according to ASTM G49 and ASTM E8 Tension test
- Align longitudinal axis of specimen (loading direction) with the Material ST grain direction

#### Loading:

- $\circ$  Loading device: Test rig ightarrow In Style of ASTM G49
- $\circ$  Loading: Constant load / displacement ightarrow according to ASTM G49
- $_{\odot}$  Load level: set at 85% Yield only ightarrow In the style ASTM G64

#### **Environment:**

- Level of Humidity: 85%  $\pm$  5%  $\rightarrow$  Ref. 1)
- Temperature: 70°C ± 1°C  $\rightarrow$  Ref. 1)

#### Test time:

 $\circ$  Test duration is recommended to be at least 100 days

#### Similar to Airbus Test Standard

HEAC Testing according to Airbus Test Standard



#### Experimental Conditions

- Specimen: 60mm round bar
- Load: Constant 85 % Rp0.2 (70 °C)
- Environment: air at different humidity levels
- Temperature: 70°C



- Initial testing to get familiar with EAC in 7085-T7452
- Using ASCOR method
  - <u>Automated Stress COrrosion Ring</u>
- Constant loading at 85% of yield strength
- 85% humidity
- 70°C
- 12 specimen
- Currently 56 days in test
  - Since September 10



## Definition of failure according to SIB

- Cracking along grain boundaries without corrosion attack or oxidation products
- If no cracking occured, check for presence of grain boundary cracking by optical examination
- SCC vs EAC fracture







Typical EAC fracture surface



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AFRL-FAA Technical Interchange Meeting on Environmentally Assisted Cracking / Stress Corrosion Cracking of High-Strength 7000 Series Aluminium Alloys Airbus Briefing

Export Control Not Listed. This Item is not listed against the EC regulations in the EU/FR

Zak Barrett, Senior Expert, Engineering Failure Analysis Dayton, OH, 4 -5<sup>th</sup> Nov 2024



#### Overview of the next 15 minutes.

- Unexpected cracking of 7xxx Alloys from the perspective of an Airframer.
  - How does Airbus define 'unexpected cracking'
  - Where do we typically see the issue?
  - What are the typical fractographic characteristics?
  - How has Airbus investigated the humid air performance of 7xxx alloys?
  - What are the key influencing factors from a service perspective?
  - How does Airbus ensure continued airworthiness?
- What are the 3-5 most critical questions that need to be answered?
  - Short term, long term.
  - The need for a collaborative approach.

[Airbus Amber]

# Unexpected cracking of 7xxx Alloys from the perspective of an Airframer.

## How does Airbus define 'Unexpected Cracking'?

Occurs in components that have:

Been manufactured from 3rd Generation 7xxx alloys that successfully passed the ASTM G47 qualification requirements.

Designed using long established design principles have produced no cracking in otherwise technically identical components manufactured from 7010\7050 in a similar temper.

Experienced cracking driven by exposure to the normal aircraft operating environment.



## Where does Airbus typically see the issue?

 Cracks typically initiate at the periphery of fastener holes and propagate in a plane perpendicular to the Short Transverse (ST) direction on the L-LT plane of the semi-finished product.



- Cracking can occur where standing stresses are present such as those induced by:
  - Build Stresses
  - Thermal effects.

Monolithic design principles used successfully for decades.

No fracture of this type has been recorded in parts manufactured from 7010\7050-T7651 plate or forgings.

## What are the typical fractographic characteristics?

Brittle mode of material separation



Gapping grain boundaries

Little or no VISIBLE evidence of material dissolution on the fracture surface.

**AIRBUS** 

7085-T7651 Plate. Fracture morphology

## The failure leaves a distinctive fractographic signature:



Intact grain boundary precipitates.

- Specific imaging conditions are required to accurately identify HEAC as failure cause:
  - SEM parameters optimised to resolve surface detail. E.g. Low Kv
  - >6000x magnification.

## How has Airbus investigated the humid air performance of 7xxx alloys?

#### Internal Test Work

Principle Aims: To understand the effect of:

- Stress
- Temperature
- Product form
- Alloy chemistry & temper

on susceptibility to cracking.

Publication Philosophy: Public Domain where possible with UoM

#### Academic Collaboration with University of

#### <u>Manchester</u>

#### The University of Manchester

MANCHESTER

Principle Aims: Detailed understanding of driving factors.

• Based on a comparative approach, 2<sup>nd</sup> vs.3<sup>rd</sup> generation alloys.

Prof. Joe Robson will present findings this afternoon.

**Publication Philosophy: Public Domain** 

#### Internal Test Work: Approach

To accelerate the phenomena by stressing material samples at elevated temperature in a humid noncondensing environment. 70°C, 85%RH selected.





### Accelerated test results - ranking materials by time to failure.



Test Conditions: 70°C 85%RH 85%TYS(0.2 Note: Measured life is dominated by initiation time.



## Lifetime of 3<sup>rd</sup> gen alloys is ~4% of 7010/7050-T7651 In-Practice: Crack initiation is more likely.

#### Accelerated test results - ranking materials by crack growth rate



Test Conditions: 70°C 85%RH 85% Note: Falling K. All alloys in the T7651 Temper except 7037- T7452



V<sub>II</sub> 3<sup>rd</sup> gen alloys ~6-20x 7050T-7651 K<sub>1HEAC</sub> at least half 7050T-7651 In-Practice: Crack will grow faster and stop at a lower K<sub>1</sub>.

## The difference in ASTM vs. humid air response:



ASTM G47 does not reliably predict the performance of 7xxx alloys in the full range of environments that they may be exposed to.

# Other learning points from humid air test work: 7050-T7651 vs 3<sup>rd</sup> Generation alloys.

- The effect of relative humidity
  - Above ~30%RH no change in time to failure observed.
- The effect of temperature
  - As temperature increases time to failure falls. The relationship is Arrhenius.
- Other effects
  - The material chemistry. Generally, a higher Zn:Mg ratio reduces humid air performance.
  - The temper. More overaging is better. A T74 is better than a T76.
  - The grain structure. The more elongated the grain structure, the worse the performance.
  - The plate thickness. Thicker plate is generally better than thinner plate.

#### In summary:

- 3<sup>rd</sup> generation alloys are much more sensitive to cracking in humid air.
- Increasing stress and temperature increases initiation and crack growth rate
  - ASTM G47 does not identify this sensitivity.

### Key learning points from service experience.

Service experience compliments laboratory findings. Increasing stress, temperature and time at temperature all increase crack initiation and growth rates

### With this understanding, how do we manage airworthiness?

- Using the understanding from laboratory alloy performance ranking tests, we have a measure of the relative risk of cracking.
  - Using the rankings, we have taken the decision to remove 3<sup>rd</sup> generation alloys from the aircraft where sustained ST stresses are present, and replace with 7010\7050.
  - Where not possible, or not required, we have undergone a component by component assessment of cracking risk.

## We ensure continued airworthiness by inspection and repair programs, reviewed and agreed with the authorities.

## Most critical needs from an airframers perspective:

#### Short Term

- Industry wide agreement that 3<sup>rd</sup> generation alloys are sensitive to cracking in humid air.
- Collaborative development of a new test method to complement G47. Reliable and quick prediction of humid air performance.

#### Long Term

 The development of physical models to enable prediction of cracking behaviour as a function of alloy chemistry, temper, stress level and operating environment.

Key Challenges: Complexity, which makes developing a reliable relationship between test performance and service performance difficult.

Open collaboration is essential to address the issue. A multi-party, government-industry collaboration is needed.

## **Final Message:**

- There is no doubt that the new generation of 7xxx alloys have an increased susceptibility to cracking in humid air relative to 7050.
- However, there are many uses of these materials where cracking does NOT occur and the full benefits of the alloy are realised.
- Therefore, use of a new generation alloy does not automatically mean you will experience HEAC cracking.
  - An assessment of risk based on the factors discussed, coupled with inspection can be a practical management strategy.



## Thank you

AFRL FAA TIM Dayton Ohio Not in Export Control List

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## ENVIRONMENTALLY ASSISTED CRACKING OF HIGH-STRENGTH 7000 SERIES ALUMINUM ALLOYS

AFRL FAA Technical Interchange Meeting (TIM) on Environmentally Assisted Cracking (EAC) / Stress Corrosion Cracking (SCC) of High Strength 7000 Series Aluminum Alloys Nov 5<sup>th</sup> 6<sup>th</sup>, 2024

Mauricio C. Cunha Principal M&P Engineer Metallic Materials



- EAC / HEAC UNDERSTANDING
- EMBRAER EXPERIENCE WITH HEAC
- FINAL COMMENTS

## EAC / HEAC UNDERSTANDING

- Environmentally Assisted Cracking (EAC): it refers to all cracking in metals that is aided by a chemical environment
- Stress corrosion cracking (SCC) and hydrogen embrittlement (HE) are types of EAC:
  - SCC (stress corrosion cracking): crack propagation driven by an anodic corrosion reaction at the crack tip
  - HE (hydrogen embrittlement): loss of a metal's bond strength due to the presence of atomic hydrogen at grain boundaries and interstitial sites in the crystal lattice
- Recent review and research papers use the term HEAC (hydrogen environmentally assisted cracking) for newer 7000 alloys prone to cracking in humid air
- Ref.:
  - Anderson, T. L. Fracture Mechanics: Fundamentals and Applications. 3 ed (2005) or 4 ed (2017)
  - De Francisco U. et al., Hydrogen environmentally assisted cracking during static loading of AA7075 and AA7449
  - Holroyd N. at al., Environment-Induced Cracking of High-Strength Al-Zn-Mg-Cu Aluminum Alloys Past, Present and Future
  - Schwarzenböck E. et al., Environmental cracking performance of new generation thick plate 7000-T7x series alloys in humid air

## HEAC UNDERSTANDING

- HEAC key influencing factors: ٠
  - Susceptilble alloy (AI-Zn-Mg-Cu alloys, high zinc/magnesium ratio, combined with low copper content) and final temper
  - Sustained stress in a plane containing ST direction
  - Aging in a typical environment (time/temperature/humidity)





Susceptible alloy and temper condition

Ref.:

- EASA SIB No.: 2018-04R2, Issued: 04 March 2021
- Hereson, H. EASA presentation: EAC, STC Workshop, 2019

Sustained stresses in the ST ٠ direction

Sustained Stresses

> Time, temperature, humidity ٠

## EMBRAER EXPERIENCE WITH HEAC

- Embraer has been investigating this phenomenon since mid 2010 's\*
  - Meetings with regulatory agencies
  - Embraer contacted aluminum suppliers / Test campaigns were set up
  - Laboratory testings performed under high humidity and high temperature
    - Tensile-bar (ASTM G49 style specimens)
    - Double Cantilever Beam (ASTM G168 style specimens)
    - Custom Interference Fit
    - Bending testing (ASTM G39 style specimens)

\* Application of susceptible alloys was being considered at that time

## FINAL COMMENTS

- The current standard of testing for SCC (ASTM G47) is not capable of detecting the HEAC
- Necessity to:
  - Development of HEAC test standartization
  - Development of alloy classification resistance to HEAC (similar to ASTM G64 ranking)



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#### **AFRL-FAA TIM on EAC/SCC of High Strength 7000 Series Aluminum Alloys**

Lockheed Martin Experiences and Efforts – EAC/SCC of 7000 Series Alloys



J B Yates, Principal Engineer

## **Outline**

- 1. C-5 Experience
  - 7075-T6 & 7049-T73 Die Forgings
  - 7085-T7452
- 2. F-35 Experience
  - Full Scale Durability Test Teardown Findings
  - EASA 2018-04R2
  - USAF Airworthiness Advisory AA-21-03
  - Engineering Investigation EI-2296
  - Fleet Findings
  - NDI Findings
  - Next Steps
  - Recommendations

 

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### **C-5 Experience: Batman Fitting**

- Original FS 2538 Fittings Under Vertical Tail Were Found to be Cracked Due to Fatigue on Multiple Aircraft Resulting in Reanalysis and Decision to Replace Existing Fittings (7075-T6 and 7049-T73) with Redesigned Fittings of 7085-T7452 Die Forgings
- Cracking was then found on some of the new 7085 fittings at new locations
  - AFRL Characterized the dominant factor for the cracking of the fittings as being due to "high installation stress resulting from shimming anomalies during installation"
  - Gaps and permanent deformation were observed after fastener removal
  - "Cracking both at the transition radius and at interference fit fasteners is intergranular at sub-grain boundaries. The fracture surface morphologies are consistent with fractures induced in 7085 material during hot humid conditions at very high applied stresses."





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## **F-35 Durability Test Teardown Findings**

- 60 findings were noted <u>during teardown</u> that grew parallel to the major loading of the part
  - NONE of these findings were detected during the test or resulted in a test stoppage.
  - 13 were detected by Bolt Hole Eddy Current but not Eddy Current Surface Scan
- Fractographic analysis was conducted on 10 of the findings
  - Fractography revealed intergranular cracking on 8 of these findings and fatigue on the remaining 2 findings.
  - Some cracks appear to be clustered in a region of the part while others are widely distributed
  - All cracks to date are in S-L or S-T orientation









## **Mitigation Using EASA Guidance and Analysis**

- EASA issued a Safety Information Bulletin (EASA SIB 2018-04R2 13SEP2018) warning of Environmentally Assisted Cracking in certain aluminum alloys (newer 7000 series alloys with high Zinc to Magnesium ratios – including 7085) due to reports of brittle cracking in aircraft components
  - Concluded that at this time this concern is not an unsafe condition
  - Recommended more stringent accelerated SCC/EAC Testing Conditions
    - 85%TYS / 70deg C / 85%RH / 100 Days

#### USAF Released Airworthiness Advisory AA-21-03

- 7085-T7452 was tested by AFRL one invalid failure in the threads at 110 days, 4 remaining coupons survived to end of test at 183 days
- Survived beyond 100 days required by EASA SIB 2018-04R2
- Sustained Stresses are to be kept below 35 ksi
- Survey of F-35 AVFEM Sustained Loads in Max Principal Direction showed that most stress levels remain below 10 ksi. A small number of locations had sustained stresses of just under 20 ksi
- Stresses for press-fit and ForceMate bushings were examined and determined to be below MMPDS SCC Threshold
- A further review using more refined sector FEMs is in work to analyze the sustained stress in the ST & SL orientations – Expected to be lower than the max principal orientation



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### **Engineering Investigation EI-2296**

- EI-2296 was conducted to investigate the cause(s) of the Intergranular Cracking Observed in the Full-Scale Durability Test Teardown.
  - Two Phases of testing were conducted, each lasting 1 year and testing a variety of configurations (surface treatments, coatings, faying surface sealants, interference levels and coating thicknesses).
  - Two Plates (7050 and 7085) were fastened together with 3 interference fit fasteners. The interference level was selected to produce ~40 ksi stress
  - These coupons were exposed to 85% RH at 70C or 50C for one year and inspected for cracking at monthly intervals
- The results indicated that the cracking was sensitive to interference level and temperature and that it initiated at the faying surface.
  - A test series which omitted faying surface sealant, and which included fuel tank coating exhibited least cracking/longest duration without cracks
- Additional testing is currently in planning



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### **Fleet Findings**

- Suspected Intergranular Cracking has been recently found on multiple fielded aircraft
  - These findings are generally too large to remove within standard rework
    limits
  - The findings are oriented in the same direction as the predominant loading rather than perpendicular to it
  - · Some holes have multiple crack indications adjacent to each other
- One finding required an engineered repair and allowed for the excision of the finding
  - Fractographic examination proved that this indication was, in fact, intergranular cracking
  - Because multiple attempts to remove the indication had been performed prior to excision, the initiation site was not available for investigation





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#### **NDI Findings**

- A review of 7085 NDI findings for machined parts revealed a number of small indications on a fraction of the parts which were readily removed by light blending
- These NDI findings were small linear indications in the ST and SL direction on curved machined surfaces.
- Since these indications occurred on only a subset of the parts, it may indicate a relationship to the chemistry or processing of the material
- This is a recent finding, and more analysis is needed to understand if it is related to the HEAC/IGC issue

### **Technical Solution: Potential Next Steps**

- Continued Collection of Data from Fleet and Manufacturing Inspections
- Identify and Quantify Sources of Stress
  - Residual Stress
  - Assembly Stress
  - Cold Work

#### Crack Arrest Analysis

- No fatigue growth observed on FSDT or Fleet findings
- What does observed crack length tell us about  $K_{ISCC}/K_{IC}$ ?

#### Risk Analysis

- How many holes inspected? vs How many IG Cracking Findings?
- How many locations splice load in ST direction?



#### **Recommendations**

- Additional Screening/Characterization Testing for New Aluminum Alloys
  - Traditional SCC Testing did not predict the HEAC/IGC Sensitivity Exhibited by 7085-T7452
- Testing for Varying Levels of Humidity/Temperature with Increased Resolution
  - Cracking exhibited on Full Scale Durability Tests did not require exposure to typical operation environments



AFRL-FAA Technical Interchange Meeting (TIM) on Environmentally Assisted Cracking (EAC) / Stress Corrosion Cracking (SCC) of High-Strength 7000 Series Aluminum Alloys November 5-6, 2024 in Dayton, OH

Ryan Davis, Wenping Zhang



## **Bounding the Problem and Key Influential Factors**

# How has your organization bounded the problem definition of unexpected cracking in 7XXX aluminum alloys in service? If possible, provide a brief explanation and examples in a pre-competitive format.

Arconic understanding is that it is a particular type of environmental assisted cracking ("EAC") known as stress-corrosion cracking ("SCC") caused by high sustained tensile stress in the normal operating environment of certain 7xxx alloy/tempers. SCC has been a known potential risk in 7xxx series aluminum alloys for more than 70 years.

# What key influencing factors are suspected for the damage mechanism(s) identified above (e.g. humidity, temperature, steady-state stress, other)?

Certain 7xxx alloys/tempers, high sustained tensile stress, environmental conditions (humidity, temperature, etc.)



## **Perceived Critical Questions and Potential Solutions**

#### What are the 3-5 most critical questions that you think need to be answered?

- 1. What mechanism(s) are most influential in development of SCC in this specific environmental condition?
- 2. Why are some materials (alloy/temper) more susceptible to this type of cracking than others?
- 3. What levers can be utilized to control the behavior of SCC (EAC) phenomena?
- 4. What is the right test to assess the susceptibility of 7xxx to this type of cracking? How to judge if a test method is good?
- 5. Will a new class of 7xxx plate products be required by OEMs or will closely monitored existing products be sufficient?

#### What are your hypotheses about the problem and potential solutions to that problem?

**Problem Statement:** literature has shown that 7xxx series alloys/tempers under high sustained tensile stresses operating in normal environmental conditions (presence of moisture, typical operating temperature, etc.) can be susceptible to stress corrosion cracking (SCC).

**Potential Solutions**: there are three elements to the SCC problem: materials, sustained tensile stress, and environment. Selecting a 7xxx/temper with adequate SCC resistance and adherence to common practices in design to reduce the sustained tensile stress to be within the material capability can be potential solutions to this problem.



## Participant Resourcing and Crowdsourcing Research

**Does your organization have the resources to answer the 3-5 questions?** Arconic has technical resources dependent on Supplier/Customer agreements.

If so, what is the timeline to address the 3-5 questions?

#### Will this information be shared with the community?

No, Arconic would consider any findings its own intellectual property.

#### How would the community validate your work so it can be used to the greatest impact?

No comment

# Do you foresee any significant challenges with translating research results into practical applications (e.g. predictive modeling or the establishment of threshold criteria)?

- Legal liability would need to be managed through appropriate Supplier/Customer contracts
- Confidential information would need to managed through NDAs, and other research/development agreements



# What suggestions does your organization have for a community-wide standardized test to address the cracking problem, and could it be used as part of the material qualification/certification process?

Arconic recognizes the adopted industry testing standards of ASTM for specific aerospace products. Arconic is open to participating in the balloting and approval process that leads to industry-wide agreement of a new test standard. A 100+ day test is likely not realistic/feasible for lot release, but it's reasonable to see this becoming a criteria for material qualification.

#### Is there a suggested path forward for new alloy development?

Depends on OEM interests for a new product and willingness to partner in the cost of development for a specific metallic solution.

# Do you have suggestions for preferred community-wide collaboration mechanisms to share research progress or engineering solutions?

No comment







Presentation for AFRL/FAA Joint Symposium Dayton, Ohio, USA November 5, 2024



# > Introduction

Constellium has been actively addressing the topic of EAC/SCC of 7xxx alloys as described in EASA SIB 2018-04 for many years

- Our understanding of this topic has been informed by:
  - > Extensive testing, characterization, and analysis at our world renowned R&D center (C-TEC)
  - > In depth analysis of the open scientific literature
  - Partnerships with best in class academic laboratories (e.g. Max Planck Institute (MPIE), Brunel University, ...)
  - > Confidential customer discussions

As a metal supplier we have no direct expertise regarding the stresses or environments that our materials are exposed to



### Problem Definition Terminology related to Environmentally Assisted Cracking

Environmentally Assisted Cracking is generally used in the industry as a generic term covering many phenomena, themselves not fully distinct from each other

 SCC cracks for 7xxx aluminum propagate via a hydrogen embrittlement mechanism in both humid air and salt water.

Corrosion reactions at the crack tip generate hydrogen atoms that can diffuse into the material and embrittle it ahead of the crack.



Diagram based on definitions in ASTM G193: Standard terminology and acronyms relating to corrosion



### **Problem Definition**

Constellium in service knowledge limited to content of EASA bulletin and bilateral exchanges

#### SIB 2018-04R2 Description

• "...caused by hydrogen embrittlement along the grain boundaries..."

"…brittle cracking…"

"...usually propagate in a plane perpendicular to the short transverse (ST) direction."

Predominantly intergranular fracture path with little or no evidence of ductile decohesion"

"…decrease of toughness but in absence of an obvious corrosion reaction…"



Figure 5 – Typical EAC fracture surface



## **Problem Definition**

Indistinguishable from reported in service SCC

Similar fracture surfaces to the "typical EAC fracture surface" have been observed in-service for 7XXX alloys and attributed to SCC

 Brittle, intergranular fracture in a plane perpendicular to the ST direction

No "obvious" corrosion visible

Selected references stating that in-service SCC often occurs without any visible macroscopic corrosion

- 1. NASA MSFC Spec 522B (NASA, 1987):
- 2. Wallace, Hoeppner, & Kandachar, <u>AGARD Corrosion Handbook</u>, volume 1, 1985
- 3. Gruhl, "Stress Corrosion Cracking of High Strength Aluminium Alloys,"1984 :
- 4. (Stress Corrosion Cracking) in Metals Handbook: Failure Analysis and Prevention, volume 11
- 5. T. D. Burleigh, Corrosion vol. 47, No. 2, (1991)

#### In-service SCC failure of 7075-T6



Fig. 7 Clearly intergraphic. SCC fracture surface: SEM fractograph from a die-forged 7075-T6 aluminium alloy engine truss mount



Image from: R.J.H. Wanhill, R.T. Byrnes, and C.L. Smith, <u>Stress</u> <u>Corrosion Cracking in Aerospace Vehicles</u>, National Aerospace Laboratory (NLR) Report Number NLR-TP-2010-538, 2010.





**Problem Definition** 

on fracture surfaces

SEM images showing CAM's on SCC fracture surface of a 7xxx alloy tested at 70°C and 85% R.H. using a double **Corrosion Products in CAMs** cantilever beam specimen



M. L. Freixes et al., "Crack arrest markings in stress corrosion cracking of 7xxx aluminium alloys: Insights into active hydrogen embrittlement mechanisms," Scripta Materialia, 237 (2023) 115690

The combination of oxidation of aluminum (corrosion) and reduction of water creates the potentially adsorbed hydrogen ions:  $2AI + 3H_2O \rightarrow AI_2O_3 + 6H^+ + 6e^-$ 

 $H_2O + e^- \rightarrow H_{ads}(c) + OH^-$ 

7xxx SCC Mechanism: Corrosion/hydrogen generation  $\rightarrow$  Embrittlement  $\rightarrow$  Propagation



### Problem Definition In-Service Stress Corrosion Cracking: Corrosion Products



• Low accelerating voltage increases the visibility of corrosion products (oxides/hydroxides) for SCC in humid air

 7xxx SCC Mechanism: Corrosion/hydrogen generation → Embrittlement → Propagation

- Crack arrest markings are associated with alternating degrees of oxidation
- Crack arrest markings on top of brittle intergranular fracture surface also observed for in-service SCC (e.g. Lynch 2017)
  - > "There was no doubt that the cracks were due to SCC..."
  - > "...intergranular facets exhibiting crack-arrest markings (CAMs)..."



Lynch, S. (2017). Failures of metallic components involving environmental degradation and material-selection issues. *Corrosion Review*.



## Key Parameters affecting SCC Resistance Material Parameters

- The same trends are observed in humid air and in ASTM G44/G47
- SCC resistance of 7xxx alloys is not solely determined by chemical composition, it is determined by a combination of parameters
- Temper: SCC Resistance T73 > T74 > T76 > T79 > T6
- Thinner plates are less resistant and fail faster
  Flatter grain structure
- Cu content has the largest compositional effect on SCC resistance
  Especially when over-aged

T. Warner et al., "Effect of Testing Conditions, Gauge, and Temper on Stress Corrosion Cracking of AA7xxx Aluminum Aerospace Plate Alloys," *Corrosion, vol. 79, issue 1, 2023, (35-47)* 



## **Key Parameters affecting SCC Resistance** Material Parameters

 Over-ageing and Cu content have larger effects than Zn:Mg ratio

- Same casting and rolling campaign, ageing performed in same furnace for same times
- Same plate thickness (4 inch / 102 mm)
- Systematic variation in Zn:Mg and Cu



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## **Key Parameters affecting SCC Resistance** Testing Parameters: Applied Stress/Sustained Stress



- > There are substantial effects of stress on SCC life-time in humid air (SIB 2018 04R1 test) and in 3.5% NaCI (ASTM G44/G47)
- Products show a typical "threshold curve" with stress level
- > The "threshold stress" depends on the environment (temperature, humidity, chloride content, etc...)

## **Key Parameters affecting SCC Resistance** Testing Parameters: Applied Stress/Sustained Stress

ASTM G64 Rating	Applied Stress Capability	Typical 7xxx products in ST direction
A	75% of specified minimum yield strength	T73 tempers
В	50% of specified minimum yield strength	T74 tempers
С	25% of specified minimum yield strength	T76 tempers
D	Fails to meet C rating	T6 tempers

It is recommended that any standardized test be flexible regarding applied stress to account for the variation in needed SCC resistance

> Example language from ASTM G47 regarding applied stress: "One or more levels as specified or as required to determine comparative stress corrosion resistance"

#### ASTM G64 or G47

Less SCC resistant/higher strength tempers are tested at lower stress

#### SIB 2018-04R2

Less SCC resistant/higher strength tempers are **tested at higher stress** 

## **Key Parameters affecting SCC Resistance** Testing Parameters: Stress and Temperature

ISO 7539-1: Corrosion of Metals and Alloys – Stress Corrosion Testing

- \* ...it is clear that the test temperature should be closely controlled and whenever possible this should be selected to correspond to that expected in service, ...increased temperature is sometimes used to accelerate test results, clearly such an approach must be undertaken with caution."
- NACE/ASTM G31: Standard Guide for Laboratory Immersion Corrosion Testing of Metals
  - If the test is to be a guide for the selection of a material for a particular purpose, the limits of the controlling factors in service shall be determined. These factors include oxygen concentration, temperature, rate of flow, pH value, composition..."
- The test temperature should correspond to the expected maximum in-service temperature

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> Avoiding activation of other mechanisms / rate determining steps

**E.g.** NASA MSFC-STD-3029A describes a test for "Stress Corrosion Susceptibility" in humid air" using 97% R.H. at  $38^{\circ}$ C (100°F)



T. Warner et al., "Effect of Testing Conditions, Gauge, and Temper on Stress Corrosion Cracking of AA7xxx Aluminum Aerospace Plate Alloys," *Corrosion, vol. 79, issue 1, 2023, (35-47)* 

12

## **Critical Questions for the Aerospace Industry**

• What are the actual temperature and sustained stress levels that 7xxx aluminum products are exposed to during service?

► Is the level of sustained tensile stress consistent with the SCC resistance of the material implied by its SCC rating (ASTM G64)?

• What is the most representative testing method to measure relative SCC resistance under humid conditions?

- > Actual in-service temperatures and sustained tensile stress should be accounted for in the test method
- Crack rate could also be relevant if there is a pre-existing crack (e.g. double cantilever beam specimens)



## Suggested Next Steps for Collaboration

- Could be divided into several topics such as:
  - > Understanding of underlying mechanisms
    - · Active participants: academic partners and industry
    - Co-funded PhDs?
  - > Development of a standardized testing practice
    - Active participants: industry, standardization organizations, (academic partners)
    - Co-funded activity within standardization organizations?
  - > Development of design / manufacturing / materials selection rules to avoid future cracking issues
    - Active participants: industry, standardization/certification organizations



For more information about stress corrosion cracking in humid air, please see Constellium's presentation to MMPDS (September 2019), our presentation to ICAA (October 2020), our presentation to Aeromat (May 2021), and our presentation to ICAA (June 2024)



## www.constellium.com





IDEAS.MATERIALIZED.

# Stress corrosion cracks propagate by hydrogen embrittlement in 7xxx aluminum alloys

SIB 2018-04R1: "...caused by hydrogen embrittlement along the grain boundaries..."

In 7xxx aluminum alloys, crack propagation during SCC is widely accepted in the scientific literature as being due to hydrogen embrittlement

- > The hydrogen is supplied via aluminum oxidation by  $H_2O$
- > Selected corrosion review papers as an example:
- T. D. Burleigh, Corrosion vol. 47, No. 2, (1991) p. 97

"...the proposed mechanism of SCC depends on the alloy system in question: anodic dissolution is favored by most researchers in the 2xxx alloys (Al-Cu and Al-Cu-Li), while hydrogen-induced cracking is favored by most researchers in the 7xxx and 5xxx alloys"

S. P. Knight et al., Corrosion Science vol. 98 (2015) p. 51

"SCC in 7xxx Al alloys is generally attributed to the embrittling effects of hydrogen, although the precise mechanisms are still being debated"

N. Ben Ali et al., Scripta Materialia vol. 65 (2011) p. 210

"The fracture of high-strength aluminum alloys, especially via stress corrosion cracking and corrosion fatigue, often involves hydrogen"

S. Lynch, Corrosion Review vol. 30 (2012), p. 72

"Mechanisms of SCC based on the effects of hydrogen on deformation and fracture are widely accepted for many materials, e.g., high-strength steels, nickel alloys, titanium alloys, aluminium alloys, and magnesium alloys,"



# The hydrogen ions are generated by a corrosion reaction

SIB 2018-04R1: "...caused by hydrogen embrittlement along the grain boundaries..."

"...absence of an obvious corrosion reaction..."

Unlike steels, aluminum alloys do not suffer from hydrogen embrittlement when exposed to (dry) hydrogen gas, even under very high pressures

The combination of oxidation of aluminum (corrosion) and reduction of water creates the potentially adsorbed hydrogen ions

J. R. Scully, G. A. Young Jr., and S. W. Smith, "Hydrogen embrittlement of aluminum and aluminum-based alloys," in Gaseous hydrogen embrittlement of materials in energy technologies, vol. 1, Philadelphia: Woodhead Publishing, 2012, pp. 707–768.

"During AI oxidation in humid air the following reaction sequence occurs spontaneously or naturally. First aluminum is oxidized in water vapor to form oxide according to the following half cell reaction:"

 $2AI + 3H_2O \rightarrow AI_2O_3 + 6H^+ + 6e^-$ 

"...In neutral waters, the following reaction occurs..."

 $H_2O + e^- \rightarrow H_{ads}(c) + OH^-$ 



## > The hydrogen ions are generated by a corrosion reaction

SIB 2018-04R1: "...absence of an obvious corrosion reaction..."

- NASA MSFC Spec 522B (NASA, 1987):
  - Service failures due to stress corrosion are frequently encountered for which the surface of the failed parts are not visibly corroded in a general sense."
- Wallace, Hoeppner, & Kandachar, AGARD Corrosion Handbook, volume 1, 1985
  - Stress corrosion is a particularly insidious form of corrosion since it will often occur with little or no visual evidence of surface corrosion such as surface discolouration or the build up of noticeable corrosion products."
- Gruhl, "Stress Corrosion Cracking of High Strength Aluminium Alloys,"1984 :
  - > "The real SCC in which frequently without the appearance of any visible corrosion attack, a discontinuous spontaneous crack formation occurs."
- (Stress Corrosion Cracking) in Metals Handbook: Failure Analysis and Prevention, volume 11
  - \* "However, the presence of staining or corrosion products on the fracture surface is by no means positive proof of SCC. Some SCC fractures are not stained or discolored, especially in materials with good corrosion resistance; in addition, many fractures become corroded before inspection can be accomplished."
- T. D. Burleigh, Corrosion vol. 47, No. 2, (1991)
  - > "During SCC, the aluminum alloy's surface often appears almost corrosion-free, ...although outwardly a structure might appear uncorroded, fine cracks could cause it to be near failure."





#### ENVIRONMENTALLY ASSISTED CRACKING

NOVEMBER 5 6, 2024 DAYTON, OH





#### EASA SIB 2018-04R2

cannot be excluded.

		EASA SIB No.: 2018-04R2	
	SA	Safety Information Bulletin Airworthiness	
coropean onion Aviation	in solety Agency	SIB No.: 2018-04R2	
		Issued: 04 March 2021	
Subject:	Environme	ntally Assisted Cracking in certain Aluminium Alloys	
Revision:			
This SIB revises EAS	A SIB 2018-04R1	dated 13 September 2018.	
Ref. Publications:			
None.			
Applicability:			
Type Certificate (TC	) holders, Supple	emental Type Certificate holders, equipment manufacturers,	
maintenance organ	isations, product	ion organisations and aluminium alloy producers.	
Description:			
EASA received repo	rts of brittle crac	king of aluminium alloy components. Additional investigation	
of some new gener	ation 7xxx series	alloys has shown that these have a sensitivity to a	
phenomenon know	n as environmen	tally assisted cracking (EAC), when subject to certain	
conditions in the no	ormal operating e	environment. The type of EAC encountered appears to be	
caused by hydroger	ation These crac	along the grain boundaries, leading to crack initiation and	
concentration and	usually propagate	e in a plane perpendicular to the short transverse (ST)	
direction. This pher	omenon has bee	en linked to the chemical composition of the alloy, notably a	
high zinc/magnesiu	m ratio, combine	ed with low copper content. Brittle fractures have been	
reproduced under I	aboratory enviro	nment and cracking has proven to be driven by time exposure	
(ageing) and is not	fatigue related, a	Ithough further crack propagation under operative loads	

**Confidential / Internal** 

EASA SIB No.: 2018-04R2

with loss of material accompanied with findings of active corrosion like pitting or attack of grain boundaries (see Appendix 1 of this SIB, Figure 6).

Sensitivity to this form of EAC has been confirmed for alloys 7037, 7040 (see Note 2 of this SIB), 7055, 7085, 7099, 7140 (see Note 2 of this SIB), and 7449. Other alloys with similar compositions might also be affected. The material temper (i.e. the specified heat treatment and additional processing such as ageing and stress relief by stretching) and product form can also influence resistance to EAC.

Note 2: Aluminium alloys 7040 and 7140 have been found to be sensitive to this form of EAC in T7651 temper, whereas the T7451 temper of these alloys has been observed to demonstrate acceptable behaviour in applications of some TC holders. Other tempers commonly used with 7040 alloys have, thus far, not exhibited sensitivity to EAC.

Occurrences of this form of EAC cannot be excluded in service and, if not detected, could lead to crack propagation, possibly resulting in reduced structural integrity. For specific designs that have already been identified, mandatory inspections and corrective actions have been initiated and further mandatory actions for other specific designs may follow.

EASA issued SIB 2018-04 to raise awareness, in all sectors of the industry, concerning this EAC phenomenon of these types of aluminium alloys. That SIB was revised to add aluminium alloy 7140 and to provide a generic test method that can be used to identify material susceptibility to this form of EAC. The current revision of this SIB provides further information on the aluminium alloy 7140 in T7451 temper.

At this time, the safety concern described in this SIB is not considered to be an unsafe condition that would warrant Airworthiness Directive (AD) action under Regulation (EU) <u>748/2012</u>, Part 21.A.3B.

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#### EASA SIB 2018-04R2: Kaiser Materials

#### Any reports of 7099 HEAC issues from customers?

- No reports from any customers for known 7099-T7651 or 7099-T7451
- No reports of any Kaiser specific products of known HEAC issues

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#### EASA SIB2018-04R2: Test Conditions

#### **Test Condition**

- Geometry: ASTM G49/E8 round smooth bar along ST orientation
- ASTM G49 Loading fixture
- Constant Load/Displacement per ASTM G49
- Load to 85% ST, YTS per ASTM G49 loading methodology
- RH=85%<u>+</u>5% and Temp = 70°C<u>+</u>1°C
- $\circ~$  Duration min 100 days, can test out to 200 days

#### **Test Evaluation**

- o Cracking along GB without corrosion attack or oxidation products on fracture surface
- Cracking along GB at 50x
- o Retest if:
  - condensation on sample, pitting, corrosion, intergranular failure (6000x) with no evidence of ductile decohesion





Non-valid test due to condensation

Figure 2 – Round smooth bar: corroded

4

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EASA SIB2018-04R2: Kaiser Testing		Cu	Mg	Zn
		0.6-1.1	1.3-2.1	7.8-9.0
EASA SIB 2018-04 R2 Conditions	7040	1.5-2.3	1.7-2.4	5.7-6.7
- EAC requires	7140	1.3-2.3	1.5-2.4	6.2-7.0
o EAC requires	7449	1.4-2.1	1.8-2.7	7.5-8.7
+ susceptible material alloy	7055	2.0-2.6	1.8-2.3	7.6-8.4
+ sustained stress in the ST direction	7085	1.3-2.0	1.2-1.8	7.0-8.0
+ ageing in a typical environment	7099	1.4-2.1	1.6-2.3	7.4-8.4

#### **Kaiser Testing Capabilities**

Discussion

KAISER

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### ARFL-FAA TIM on HEAC in 7xxx-Series Aluminum Alloys

Justin Lamb PhD Director of Metallurgy and R&D

> Dayton, OH 5 November 2024



HEAC

#### Introduction

- Universal Alloy Corporation (UAC) has studied HEAC since the EASA SIB bulletins.
  - Focus has been placed on the following:
    - 1. Understanding the underlying mechanism published research and internal testing.
    - 2. Understanding the nuances of the proposed test mechanism in the SIB.
    - 3. Running tests on various alloy-tempers at different stress levels.
- Hydrogen embrittlement is well known to be an underlying mechanism in classic EAC (SCC, etc.) along with anodic dissolution.
  - Ex. the diffusion of hydrogen to crack tips propagating cracks.
  - HEAC appears to come about from a combination of humidity level, exposure temperature, and stress.
    - The expectation is that like SCC chemical composition, temper, and grain structure also play a role in a component's susceptibility.



HEAC

#### HEAC Critical Questions





- What is the role of temperature on HEAC?
  - Aluminum alloys tend to lose strength and experience modulus changes with temperature (ex. MMPDS 7075).
  - Designing for HEAC tends to push one toward T-phase 7xxx-series precipitates instead of η.
    - T-phase is generally avoided in modern alloy design due to issues with SCC and facture toughness.
    - However, T-phase may be better in thermal exposure environments.

#### HEAC Critical Questions (II)



- What is the role of temper on HEAC?
  - The EASA SIB hints that temper may play a crucial role in HEAC resistance (ref. 7040 note).
  - Issue alloys appear to be largely underaged when plotted versus equivalent time.
  - With traditional SCC in 7xxx-series aluminum alloy, corrosion resistance increased with over-aging.



#### HEAC Testing EASA SIB and Possible Alternatives



Vasudevan and Suresh (1982)

- The EASA SIB details a "EAC Generic Test Method."
  - The test appears to focus on initiation of EAC.
  - The constant strain test detailed in the EASA SIB has several disadvantages, including duration (recommendation 100 days), a high degree of sensitivity, and the need for advanced characterization techniques (SEM) per test.
- Fatigue Crack Growth Rate (FCGR) tests are known to show sensitivity to the test environment.
  - Crack growth rates in high humidity environments (>90%) are generally faster than in lab air for the same material.
    - Reported mechanisms are similar to those reported for HEAC → could a FCGR test be developed to more easily evaluate materials?



HEAC

# Problem Definition and Knowledge Gaps for 7xxx EAC

### J.T. Burns, R.G. Kelly

University of Virginia

### Z.D. Harris

University of Pittsburgh

#### AFRL-FAA Technical Interchange Meeting (TIM) on Environmentally Assisted Cracking (EAC) / Stress Corrosion Cracking (SCC) of High-Strength 7000 Series Aluminum Alloys

November 2024 Dayton, OH





# Damage Mode

Crack formation and stable growth below K<sub>IC</sub> caused by timedependent damage involving the conjoint interaction of.....







### Step 1: Cracking "pre-cursor"



Harlow, Int J Fat, 2010



#### Step 1: Cracking "pre-cursor"



In the absence of environmental degradation surface defects and/or constituent particles often serve as crack nucleation sites...







#### Step 1: Cracking "pre-cursor"



#### These constituent particles are also sites of localized corrosion in the presence of an electrolyte

Table III. Pitting potentials for intermetallic compounds common in aluminum alloys. Results are average values incorporating the results of numerous tests.

		Pitting potential (mV <sub>SCE</sub> )			
Stoichiometry	Phase	0.01 M	0.1 M	0.6 M	Note
Al <sub>3</sub> Fe	β	442	106	-382	
Al <sub>2</sub> Cu	θ	-434	-544	-652	
Al <sub>3</sub> Zr	β	-223	-275	-346	
Al <sub>6</sub> Mn	-	-485	-755	-778	
Al <sub>3</sub> Ti	β	-232	-225	-646	
Al <sub>32</sub> Zn <sub>49</sub>	Ť'	-	-	-	С
Mg <sub>2</sub> Al <sub>3</sub>	β	-818	-846	-959	
MgZn <sub>2</sub>	Μ, η	-	-	-	С
Mg <sub>2</sub> Si	β	-	-	-	С
Al <sub>7</sub> Cu <sub>2</sub> Fe	-	-447	-448	-580	
Mg (AlCu)	-	224	-2	-	D, E
Al <sub>2</sub> CuMg	S	108	80	135	F
Al <sub>20</sub> Cu <sub>2</sub> Mn <sub>3</sub>	-	-210	-428	-534	
Al <sub>12</sub> Mn <sub>3</sub> Si	-	-563	-621	-712	
A1 (99.9999)	-	-545	-610	-696	Α
Cu (99.9)	-	19	-30	-94	Α
Si (99.9995)	α	-	-	-	A, C
Mg (99.9)	-	-1095	-1391	-1473	A, G
Mn (99.9)	-	-	-	-	A, C
Cr (99.0)	-	479	297	190	Α
Zn (99.99)	-	-	-	-	A, C
Al-2%Cu	α	-447	-471	-529	В
Al-4%Cu	α	-418	-406	-465	в
7X75 Matrix	-	-633	-736	-768	Μ
AA 7075-T651	-	-684	-739	-810	х







### Step 1: Cracking "pre-cursor"

80 µm



Localized corrosion leads to local stress concentrators and local H uptake



(c) Localized corrosion mechanism in 3.5% NaCl solution environment





**Step 2: Crack Initiation** 





# **Step 2: Crack Initiation**

# Fundamentally requires the local exceedance of a critical stress/strain value on a microstructural feature

#### Typically Aided By:

- Microstructural interface with a low cohesive strength
- Local stress/strain concentrators (pit, IG fissure, constituent, HA grain interface, etc)
- H-based degradation of the cohesive strength of a microstructural interface
- Aggressive local chemistry (due to pit IG, fissure, crevice, etc)





### **Step 2: Crack Initiation**

#### Corrosion damage influences crack formation by... Local Environment Aggressive Local Environment Concurrent loading/dissolution **Compsion Fatigue** Nucleation Local Embrittlement Threshold Enhanced aggressive environment H-charging from prewithin pit Nominal existing corrosion Environme embrittles material and reduces cohesion Nominal Enhanced Stress/Strain Local Material Susceptibility Global due to decreased cross-sectional area Local Nominal Material Susceptibility Macro- and micro-stress/strain Initial Conditions at Corrosion Faligue concentrators Initiation Site Schmidt, 1996 Local microstructure I neal Strees DOT/FAA/AR-95-108 features (constituents, Figure 1: Critical conditions for corrosion-fatigue crack nucleation.



etc.)



## **Step 2: Crack Initiation**

# Be aware, but wary of engineering scale (LEFM) models of crack initiation (e.g. Kondo approach)

- Generally, assume a pit is a crack; when pit size and stress result in K exceeding K<sub>ISCC</sub>, then initiation...
- Issues with (1) violating LEFM crack size assumptions and (2) small cracks through a few grains will not have continuum behavior
- Rigorous modeling would required grain scale driving forces and failure criteria (akin to FIPs for fatigue)





### Step 3: Small Crack Growth

### **Both:**

- Chemically small
- Mechanically small
  - Where cracking through a few grains

•	SHORT CRACK	MICROSTRUCTURAL		
•	•r <sub>pc</sub> >>gs •Many grains & front	• In 1 to 5 grains		
MECHANICAL	SMALL SCALE YIELDING NOT MAINTAINED	ENHANCED CRACK TIP STRAIN		
	• High σ <sub>NET</sub>	• a < gs		
	$a < 1 lo 5 r_p$	• f <sub>pc</sub> < \$ to 2 gs		
PHYSICAL	REDUCED CRACK WAKE CLOSURE			
	• a < 1 mm			
CHEMICAL	CRACK SIZE DEPENDENT			
	OCCLUDED CHEMISTRY			
	+ a < 5 to 10 mm			
	$f_p = monotonic plastic zone  f_{pc} = cyclic plastic zone$	a - crack length gs - grain size		





### Step 3: Small Crack Growth

#### **Both:**

- Chemically small
- Mechanically small
  - Where cracking through a few grains
  - Results in a lot of variability and difficult to characterize







## Step 4: Continuum scale crack growth

- Crack is interacting with a large number of grains and you get continuum scale behavior (LEFM will work)
  - $da/dt = f(K); K = \sigma \sqrt{\pi a} (\beta)$
- Well established techniques for generating da/dt vs K data (ISO standard available, improved ASTM standard underway)
  - Able to pull out continuum scale threshold and Stage II kinetics
- Can generate data for relevant/bounding environments; important to ensure environmental similitude







#### Step 4: Continuum scale crack growth

#### Commercial software exists to enable LEFM-based EAC life prediction

- Akin to AFGROW, NASGROW, BEASY for fatigue
- Simplistically, integration of da/dt = f(K); can add probablistics



SCCrack Developed by B. Tryon, VEXTEC

Probabilistic Fracture Mechanics Simulation of Stress Corrosion Cracking Using Accelerated Laboratory Testing and Multi-Scale Modeling

Richard P. Gangloff<sup>1</sup>

ENVIRONMENTALLY ASSISTED CRACKING

#### ABSTRACT

A propagation approach to manage stress corrosion cracking A propagation approach to marage stress correction cracking (SCC) is justified for modern high-strength alloys that exhibit low-subcriticed crack growth rates, and is enabled by ma-turing probabilistic fracture mechaniss and mechanism-based hydrogen damage madeling. A computer program, SCCnack, is developed based on stress intensity (R) similitate and fast So developed vision of stress indecorp in strature and p Monte Carlo analysis to predict SCC [ife distributions from input distributions of variable material crack growth rate (da/dt vs. K), starting crack size, environment, and stress. Growth rates are obtained using an accelerated experiment that Grotein rules we obtained using a functionennum appennum run exploits precision electrical potential measurement of crack propagation, small crack size, and slow-rising K loading. Data are levenged by fundamental models of SOC threshold (K<sub>y</sub>) and H «Iffusion limited Stage II da/dt, built on the hydrogen environment assisted cracking mechanism. With these inputs, SCCrack enables multi-scale "What if?" simulations of the effects of alloy-environment-precorrosion stress variat on component cracking. Examples are presented for SOC in modern ultra-high strength martensitic steel (AerMet<sup>ra</sup>100 UNS K92580 and Ferrium<sup>18</sup>M54 UNS K91973), a Ni-Cu UNA 6822000 mia rentamination (UNA 681973), and a superalay Monet K500 (UNA 805500), and a sensitized AEMg alay (Alay 5063-H131 (UNA 806083), each stressed in cloride solution with varying antibolic polectation. Research is required to enhance accelerated measurement of very low da/dt, to capture the effects of atmospheric-environment spectra, to advance mechanism modeling, and to validate the approach for field-specific components.

submitted for publication: September 30, 2015, Revised an incommercian parameteric operations of 2010 to the operation of 2010 to the operation of the operation of

Science and Engi ISSN 0010-9312 (print), 1938-159X (online) 00125/\$5.00+\$0.500 © 2016, NACE International

CORROSION-JULY 2016

KEY WORDS: aluminum allou, fracture mechanics, hudroger KEY WORLD: building on environment assisted cracking mechanism, nickel-copper alloys, predictive calculations, steel,

In contrast to quantitative-sophisticated fatigue crack growth prognosis methodologies embodied in widely used fracture mechanics and micromechanics

programs,1-3 control of stress corrosion cracking (SCC)

is largely qualitative and based on using highly resistant alloys for service in aggressive environments. Material selection and environment chemistry control

typically rely on smooth-specimen data often deter

justified by the assumptions that SCC is initiation controlled and components are defect free. If crack

mined with a mechanical or chemical accelerant,4 as

propagation is considered, then SCC control is often based on a go or no-go threshold stress intensity factor, K<sub>TB</sub>, and assumed infinite-life design.<sup>5-6</sup> T

approach stemmed from the high rates of subcritical SCC propagation, reported as a crack growth rate

(da/dt) vs. stress intensity (K) relationship, for hightrength alloys in acrospace and marine e In contrast, a damage tolerant method effectively

predicts SCC in low-strength C-Mn and austenitic stainless steels stressed in nuclear reactor environ

The SOC resistance of high-strength-high-toughness Ti-, Fe-, Ni-, and Al-based alloys has been substantially improved by composition and therm mechanical processing, as well as by coating and

ments and typified by very low da/dt."

sion cracking

NTRODUCTION



INIVERSITY

### **Step 4: Continuum scale crack growth**

- Commercial software exists to enable LEFM-based EAC life prediction
  - Akin to AFGROW, NASGROW, BEASY for fatigue
  - Simplistically, integration of da/dt = f(K); can add probablistics

Can be very accurate, but predictions will only be as good as the material/environment property (da/dt vs K) and accuracy of stress input



SCCrack Developed by B. Tryon, VEXTEC

#### Richard P. Gangloff<sup>1</sup>

ABSTRACT

A propagation supported in transmiss stress correction reacking ISCC1 is stretified to reach might strength of long-that orbits linesubstritted or rank growth reaches, and is a malibility in using probability. Explanme machines and mechanism-based haldways damage makeling. A surgeator program. SC Crack, Monte Carlos concepts to prodect SC CI discubstants from linguit attratistical or solution of the strength of the linguity of the strength of the strength of the linguity of the strength of the strength of the strength input distributions of south strength of the strength of linguity of the strength of the strength of the strength of the linguity of the strength of the strength of the strength of the linguity of the strength of the strength of the strength of the linguity of the strength of the strength of the strength of the linguity of the strength of the strength of the strength of the linguity of the strength of the strength of the strength of the linguity of the strength of the linguity of the strength on strength of the strength of the linguity of the strength on strength of the strength of the strength of the strength on strength of the strength of the strength of the strength on strength of the strength of the strength of the strength on strength of the strength of the strength of the strength on strength of the strength of the strength of the strength on strength of the strength of the strength of the strength of the strength on strength of the strength of the strength of the strength on strength of the strength of the strength of the strength on strength of the strength of the strength of the strength on strength on strength of the strength of the strength of the strength on strength on strength on strength on specific to a strength on strength on strength on strength on strength on specific to a strength on strength on strength on strength on strength on specific to a strength on strength on strength on str

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ment of Materials Science and Engineering, University of Vinginia, Charlotteeville, VA. ISSN 0010-8012 (print), 1938-159X (online) 16/00012555.00-50.500 © 2016. NACE Internet tona

RST WORDS: nhumhum allag, fracture mechanics. hydrogen ambritianent, hydrogen ensionment assisted cracking mechanism, nickel-copper allays, predictive colculations, steel, stress corrosion cracking.

#### NTRODUCTION

In contrast to quantitative-sophisticated fatigue crack growth prognosis methodologies embodied in widely used fracture mechanics and micromechanics programs,1-3 control of stress corrosion cracking (SCC) is largely qualitative and based on using highly resistant alloys for service in aggressive environments. Material selection and environment chemistry control typically rely on smooth-specimen data often deter mined with a mechanical or chemical accelerant,4 as justified by the assumptions that SCC is initiation controlled and components are defect free. If crack propagation is considered, then SCC control is often based on a go or no-go threshold stress intensity factor, K<sub>TB</sub>, and assumed infinite-life design.<sup>5-6</sup> T approach stemmed from the high rates of subcritical SCC propagation, reported as a crack growth rate (da/dt) vs. stress intensity (K) relationship, for hightrength alloys in acrospace and marine en In contrast, a damage tolerant method effectively predicts SCC in low-strength C-Mn and austenitic stainless steels stressed in nuclear reactor environ ments and typified by very low da/dt.1

ments and typfied by very low da/dt." The SCC resistance of high-strength-hightougfmess Tr. Fe-, Ni-, and Al-based alloys has been substantially improved by composition and thermomechanical processing, as well as by coating and

CORROSION-JULY 2016



NIVERSITY



# **Step 5: Crack growth until exceedance of** $a_{crit}$ **(e.g.** failure)





# Total Life =

**Time for Step 1: Precursor development** 

- + Time for Step 2: Crack initiation
  - + Time for Step 3: Small crack growth
    - + Time for Step 4: Long crack growth





# Total Life =

**Time for Step 1: Precursor development** 

- + Time for Step 2: Crack initiation
  - + Time for Step 3: Small crack growth
    - + Time for Step 4: Long crack growth

It is important to remember this when considering testing approaches....









# **Prominent SCC/EAC Mechanisms are:**

- Stress-assisted anodic dissolution
- Film Rupture-based
- Hydrogen Embrittlement





#### Mechanism Crack Tip Passivation + Film Rupture

EAC can occur in metal-environment systems where crack wall passivation limits metal dissolution to maintain a sharp crack tip



Crack tip plastic strain breaks passive oxide, then local metal dissolution and <u>repassivation</u> occur for crack growth, ∆a

#### **Process repeats**

1. Anderson, 2005







#### Mechanism Hydrogen Environment Assisted Cracking

Many gaseous and aqueous environments react with Fe, Ni, Al, Ti alloys to produce atomic hydrogen (Hadsorbed) on the crack tip surface

Atomic H embrittles most metals by several mechanisms discussed in the next lecture







Mechanism Hydrogen Environment Assisted Cracking

## Substantial literature supporting an important role of H-embrittlement 7xxx-series EAC

Thompson AW, Mueller MP, Bernstein IM. Stress-corrosion cracking in equiaxed 7075 aluminum under tension and torsion loading. Metall Trans A 1993;24:2569–75.

Gruhl W. Stress corrosion cracking of high strength aluminum alloys. Zeitschrift Fur Met 1984;75:819–26.

Cooper KR, Young LM, Gangloff RP, Kelly RG. Electrode potential dependence of environment-assisted cracking of AA 7050. Mater Sci Forum 2000;331–337:1625–34.

Young GA, Scully JR. The effects of test temperature, temper, and alloyed copper on the hydrogen-controlled crack growth rate of an Al-Zn-Mg-(Cu) alloy (Metallurgical And Materials Transactions A (January 2002) 33A (101-115)). Metall Mater Trans A Phys Metall Mater Sci 2002;33:1167–81.

#### See Burns/Harris 7xxx EAC review for all 663 references 🙂

5. Gangloff, 2003







# Where does H come from??

to form  $H_2$  gas. During Al oxidation in humid air the following reaction sequence occurs spontaneously or naturally. First aluminum is oxidized in water vapor to form oxide according to the following half cell reaction:

$$2A1 + 3H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^-$$
 [19.2]

In acid water, the following half cell reaction must occur 6 times in order to support Al oxidation to Al(III) as indicated in Equation 19.2:

$$\mathrm{H}^{+} + \mathrm{e}^{-} \to \mathrm{H}_{\mathrm{ads}}(\mathrm{c})$$
[19.3]

In neutral waters, the following reaction occurs instead of Eq. 19.3:

$$H_2O + e^- \rightarrow H_{ads}(c) + OH^-$$
[19.4]











Ritchie-Knott-Rice (RKR)-type damage model can be used to understand the initiation, small crack growth and continuum scale growth
















### Damage Mechanism

#### Can move to a "Strain Controlled" Failure if ENVIRONMENT is less severe or the MATERIAL is more resistant...







### Damage Mechanism

Can move to a "Strain Controlled" Failure if ENVIRONMENT is less severe or the MATERIAL is more resistant...



Strain-control results in loading rate dependence...





Well-known phenomenological sensitivities can be rationalized in this paradigm

### Sensitivities of 7xxx EAC:

- Aging
- Composition
- GB PPCs and segregation
- Re-crystallization
- Plate thickness
- Polarization dependence
- Bulk electrolyte chemistry dependence
- Water content
- Temperature





Well-known phenomenological sensitivities can be rationalized in this paradigm

# 1. Modify C<sub>H</sub>

#### **Material**

- GB composition (changes the crack tip chemistry)
- H-uptake behavior
- Oxide stability
- Re-passivation behavior
- Diffusivity







Well-known phenomenological sensitivities can be rationalized in this paradigm

# 1. Modify C<sub>H</sub>

### **Material**

- GB composition (changes the crack tip chemistry)
- H-uptake behavior
- Oxide stability
- Re-passivation behavior
- Diffusivity

### Environment

- Polarization
- Electrolyte composition
- Humid air water content
- Temperature







Well-known phenomenological sensitivities can be rationalized in this paradigm

# **2. Modify** $\sigma_{yy}$

### **Material**

- Aging/Plate thickness
  - Slip planarity
  - Hardening behavior
- Tortuous crack path (lower macro-scale K)
- GB PPCs
- PFZ

### Environment

- Temperature







Well-known phenomenological sensitivities can be rationalized in this paradigm







Well-known phenomenological sensitivities can be rationalized in this paradigm

# 4. Modify $\sigma_f(C_H)$

#### **Material**

- Aging/Plate thickness
- GB PPC spacing/geometry
- GB segregation
- PFZs

### Environment

- N/A







Well-known phenomenological sensitivities can be rationalized in this paradigm













### Characteristics of a Good EAC Test:

- Clear definition of testing goals
  - Screening? Qualification? Modeling? Mechanistic understanding?
- If transferability to application is desired
  - Environmental similitude
    - Or purposely selected bounding environment
  - Mechanical similitude (LEFM)
    - Does NOT exist for "bulk stress thresholds"
- Careful control/monitoring of environment
- Isolation of variables
  - Metallurgical, environmental mechanical





#### - Legacy (Total Life)

- Qualification/Screening
- ASTM G129 (SSRT) and G49 (Tensile)
- ASTM G44 and G47 (3.5% NaCl, Alt Immersion)
- Criteria: Failure/damage observation/reduction on ductility
- Any "threshold" has no mechanical/environmental similitude!!!

### - EASA SIB 2018-04R2 (Total Life)

- Screening
- ASTM G49 (70C, 85% RH, 85% YS)
- Criteria: damage observation

### - LEFM Testing (Crack Propagation)

- ASTM E1681, G168, etc.





#### - Legacy (Total Life)

- Qualification/Screening
- ASTM G129 (SSRT) and G49 (Tensile)
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- Criteria: damage observation
- LEFM Testing (Crack Propagation)
  - ASTM E1681, G168, etc.

### Note: Details of the LEFM testing MATTERS!!!!





LEFM EAC behavior is known to depend on loading rate, however current LEFM testing do not control the loading rate







#### - ASTM EAC Testing Standard is Being Developed...

 Rising K, active crack length monitoring (dcPD), environmental flexibility, testing design to conform to LEFM constraints

Fracture mechanics-based method for assessing EAC behavior

Utilize dcPD-based crack length measurements to enable K-controllea testing of specimens exposed to an applied potential while immersed in 0.6 M NaCl







#### ASTM EAC Testing Standard is Being Developed...

- Explored for a wide-range of materials and environments
  - Strength, Oxide stability, Mechanism (HE vs AD), Toughness, Diffusivity

Alloy Class	Pertinent Material Properties Coupled corrosion-hydrogen mechanism, modest strength, slow H diffusivity		
AA7075-T651			
AA5456-H116	Coupled corrosion-hydrogen mechanism, low strength, slow H diffus		
Monel K-500	No oxide under in-service conditions, moderate strength, slow H diffusiv		
C465/Pyrowear	Very high strength, modest H diffusivity		
Beta-C <u>Ti</u>	a-C Ti Strong passive film, high strength, fast H diffusivity		
316L AD/HE mechanism, low strength, slow H diffusivity			







- ASTM EAC Testing Standard is Being Developed...

#### Take-Away

- In all cases rising-K (dK/dt > 0 MPaVm) is:
  - CONSERVATIVE
  - More Efficient (done in <1 week)</li>

ASTM E1681		Pertinent Publications		
steels ( <sub>YS</sub> < 1,200 MPa) steels ( <sub>YS</sub> > 1,200 MPa) aluminum alloys titanium alloys	10 000 hours 5 000 hours 10 000 hours 1 000 hours	<ul> <li>Monel K-500:         <ul> <li>Z.D. Harris, E.M. Dubas, A.S Popernack, B.P. Somerday, and J.T. Burns, "Elucidating the loading rate dependence of hydrogen environment-assisted cracking in a Ni-Cu superalloy", <i>Theoretical and Applied Fracture Mechanics</i>, p. 102846, 2021.</li> </ul> </li> <li><b>7xxx-Al:</b> <ul> <li>Z.D. Harris and J.T. Burns, "The effect of loading rate on the environment-assisted cracking behavior of AA7075-T651 in aqueous NaCl solution", <i>Corrosion and Materials Degradation</i>, vol. 2, p. 360-375, 2021.</li> </ul> </li> <li><b>5xxx-Al:</b> <ul> <li>Z.D. Harris and J.T. Burns, "On the loading rate dependence of environment-assisted cracking of sensitized AA5456-H116 exposed to marine environments", <i>Corrosion Science</i>, vol. 201, p. 110267, 2022.</li> <li><b>High Strength Steel:</b> <ul> <li>Z.D. Harris, R.S. Marshall, R.G. Kelly, and J.T. Burns, "Coupling Fracture Mechanics Experiments and Electrochemical Modeling to Mitigate Environment-Assisted Cracking in Engineering Components," <i>Corrosion Journal</i>, <i>79</i>(<i>3</i>), <i>p.</i> 363-375, 2023.</li> <li>R. S. Marshall, Z. D. Harris, M. K. Small, K. L. Brunner, J. T. Burns, R. G. Kelly, "A Materials Selection Framework for Fastener-in-Panel Geometries Using FEM and LEFM, to Mitigate Coating Degradation and Hydrogen Embrittlement", <i>Corrosion Science</i>, in review.</li> <li><b>Austenitic Stainless Steel:</b></li></ul></li></ul></li></ul>		
		7.D. Marrie and LT. Durns, "Evoluation of static induced estimate in gradulength measurements via the direct surrout notantial differences		

- Z.D. Harris and J.T. Burns, "Evaluation of strain-induced artifacts in crack length measurements via the direct current potential difference technique", in ASTM STP 1638: Fifth Symposium on Evaluation of Existing and New Sensor Technologies for Fatigue, Fracture, and Mechanical Testing, P. McKeighan, ed., 138–159. <u>http://doi.org/10.1520/STP163820210046</u>, 2022.
- In preparation:
  - Beta Titanium
  - Custom 465





### Why did legacy screening approaches not capture this EAC issue with 3<sup>rd</sup> generation alloys?





- Issue?
  - Not the right combination of accelerating factors for some alloys and some structures (Example: ASTM G34 for AI-Li)







- Issue?
  - Total Life Approach...







- Issue?

- Total Life Approach...

# Total Life =

**Time for Step 1: Precursor development** 

- + Time for Step 2: Crack initiation
  - + Time for Step 3: Small crack growth
    - + Time for Step 4: Long crack growth





# - Issue? - Total Life Approach...



#### Total Life =

Time for Step 1: Precursor development

- + Time for Step 2: Crack initiation
  - + Time for Step 3: Small crack growth
    - + Time for Step 4: Long crack growth

In a "Total Life" NaCl test the pre-cursor development and crack initiation is from corrosion at constituent particles





### - Issue?

#### - Total Life Approach...



7050	7085	
Metric	Metric	
87.3 - 90.3 %	87.62 - 90.42 %	
<= 0.04 %	<= 0.04 %	
2.0 - 2.6 %	1.3 - 2.0 %	
<= 0.15 %	<= 0.08 %	
1.9 - 2.6 %	1.2 - 1.8 %	
<= 0.10 %	<= 0.04 %	
<= 0.05 %	<= 0.05 %	
<= 0.15 %	<= 0.15 %	
<= 0.12 %	<= 0.06 %	
<= 0.06 %	<= 0.06 %	
5.7 - 6.7 %	7.0 - 8.0 %	
0.08 - 0.15 %	0.08 - 0.15 %	
	<b>7050</b> <b>Metric</b> 87.3 - 90.3 % <= 0.04 % 2.0 - 2.6 % <= 0.15 % 1.9 - 2.6 % <= 0.10 % <= 0.05 % <= 0.15 % <= 0.12 % <= 0.06 % 5.7 - 6.7 % 0.08 - 0.15 %	

#### Total Life =

Time for Step 1: Precursor development

- + Time for Step 2: Crack initiation
  - + Time for Step 3: Small crack growth
    - + Time for Step 4: Long crack growth

7085 would be expected to have fewer constituent particles due to composition; thus slower Step 1 and 2





Issue?
 Total Life Approx



#### Total Life Approach...

#### Total Life =

Time for Step 1: Precursor development

- + Time for Step 2: Crack initiation
  - + Time for Step 3: Small crack growth
    - + Time for Step 4: Long crack growth

The slower development of Step 1 and 2 could have masked the faster crack growth (Step 3)

Fig. 7. Decreasing stress intensity-growth rate (v-K) curves from the DCB tests conducted on the new-gen alloys, compared to benchmark data for AA7050-17651 (tested at 70 °C and 85 % RH) presented as; (a) a conventional logarithmic and (b) a linear v - K plot. Overlaid data is shown for 4 tests from each material (apart from AA7449-17651; 3 tests). The materials tested were: AA7050-17651, 140 mm plate; AA7085-17651, 150 mm plate, AA7449-17651, 95 mm plate, AA7037-17452, 136 mm forged plate.





Total Life =

### What about Zak Barrett's Airbus data showing faster initiation in 85% RH, 70C?

In a humid environment pitting at constituents may not be the pre-cursor for crack initiation so the "cleaner" alloy may be less relevant than in ASTM G47...

#### masked the faster crack growth (Step 3)

Fig. 7. Decreasing stress intensity-growth rate (v-K) curves from the DCB tests conducted on the new-gen alloys, compared to benchmark data for AA7050-17651 (tested at 70 °C and 85 % RH) presented as; (a) a conventional logarithmic and (b) a linear v - K plot. Overlaid data is shown for 4 tests from each material (apart from AA7449-17651; 3 tests). The materials tested were: AA7050-17651, 140 mm plate; AA7085-17651, 150 mm plate, AA7449-17651, 95 mm plate, AA7037-17452, 136 mm forged plate.

20

25

15

K (MPavm)

10



lssue?

(a) 1

V (ms<sup>-1</sup>)

1.0E-11

Schwarzenbock, Corr Sci, 2020



arowth



Fig. 7. Decreasing stress intensity-growth rate (v-K) curves from the DCB tests conducted on the new-gen alloys, compared to benchmark data for AA7050-17651 (tested at 70 °C and 85 % RH) presented as; (a) a conventional logarithmic and (b) a linear v - K plot. Overlaid data is shown for 4 tests from each material (apart from AA7449-17651; 3 tests). The materials tested were: AA7050-17651, 140 mm plate; AA7085-17651, 150 mm plate, AA7449-17651, 95 mm plate, AA7037-17452, 136 mm forged plate.



Schwarzenbock, Corr Sci, 2020









#### Testing Improvement

- Total life approach may mask important sensitivities
- Ensure capturing water content rather than RH
- Target relevant mechanism with testing
- Ensure testing approach matches goals (screening, mechanistic, etc)
- Employ active loading rate for LEFM testing





### Testing Improvement

- Total life approach may mask important sensitivities
- Ensure capturing water content rather than RH
- Target relevant mechanism with testing
- Ensure testing approach matches goals (screening, mechanistic, etc)
- Employ active loading rate for LEFM testing
- Work to be done:
  - Quantitatively establish the issues beyond hypotheses
  - Do we have the solution...learn from Fatigue!!!!!
    - Exposure (G44/47) for initiation/MSC
    - · LEFM characterization of growth
    - . \$1M Question: What environment???





- Engineering definition of environmental bounds
  - Reduce the research space to targeted environments and temps
  - Work to be done:
    - . Input from users
    - Identify relevant, differentiating, bounding cases; do not be paralyzed capturing every condition. Just the right ones...
    - Develop a correlation between the environment and the H-uptake





- Understanding the relative impact of the precursor, initiation, MSC, and long crack growth
  - . For both testing and for service life
  - Work to be done:
    - Current research with AFRL targeted toward this







- Mechanistic understanding beyond phenomenology for propagation to enable management/material improvement
  - Identify microstructure and environment sensitivities for propagation







- Mechanistic understanding beyond phenomenology for propagation to enable management/material improvement
  - Identify microstructure and environment sensitivities for propagation
  - Work to be done:
    - Prioritization of critical parameters that are governing the damage mechanism
      - Identify how those parameters will vary with common material specification and processing routes that are used (overage, composition, etc)
    - Work moving forward on this...
      - Thus far water content is more relevant than T





- Generation of propagation data to enable engineering management
  - What is needed/desired by the OEM and structural management community?
    - New material?
    - . LEFM based data?
    - Mitigation strategies?







- Generation of propagation data to enable engineering management
  - What is needed/desired by the OEM and structural management community?
    - · New material?
    - . LEFM based data?
    - Mitigation strategies?
  - Work to be done:
    - Generation of relevant data for materials and environments that will help with life management
    - Exercising LEFM based models





# UVa Capabilities

- · Electrochemistry expertise and equipment; Characterization facility
- EAC testing capability and expertise
  - Full range of total life and LEFM testing
  - Full range of environmental control for elevated temp and humidity control
- Experience linking EAC
   behavior to mechanism and
   causal microstructure feature
- Extensive experience with EAC in 7xxx alloys
- Ability to explore H-metal interactions






### **UVa Capabilities**

Mechanical Testing and Characterization Capabilities at University of Pittsburgh

#### **Mechanical Testing**

- 10 mechanical load frames with ability to run compression, tension, fatigue, fatigue crack growth, fracture toughness, SSRT, and SCC experiments
- dcPD systems for high-fidelity crack length monitoring under wide range of environmental conditions
- 2 Charpy systems (full size and subsize)
- Gleeble 3500 GTC plus access to another Gleeble 3500 with custom high T kit for metallurgy studies

#### **Materials Characterization**

- 1 uCT with upgraded CMOS detector
- 3 XRDs
- 3 SEMs with upgraded EDS/EBSD
- 1 EPMA
- 1 FIB (with EDS/EBSD)
- 3 TEMs (environmental, metallurgy, and probe-corrected)
- 3 digital microscopes (including one with on-board LIBS)
- And others...

Experience leveraging these tools to establish mechanistic understanding of EAC in numerous materials, including 7xxx-series Al alloys





# Example







#### Metal-rich primer-based coatings that polarize the substrate has been demonstrated to be effective in mitigating CORROSION







# Metal-rich primer-based coatings that polarize the substrate has been demonstrated to be effective in mitigating and EAC

#### **Example for 5xxx Al**



0.6 M NaCl or 5.45 M (saturated) NaCl



Burns, McMahon, 2021

# Metal-rich primer-based coatings that polarize the substrate has been demonstrated to be effective in mitigating and EAC

#### **Example for 5xxx Al**



Stress Intensity [MPa√m]

#### Vast Reduction in EAC Susceptibility due to "cathodic protection"



#### Some evidence that polarization via an MRP would be effective but very limited data for environments relevant to aerospace applications



ity and stress corrosion crack velocity in Al alloys.<sup>25</sup>



#### Talk Objectives:

- Systematically characterize the EAC behavior of 7075-T651 and 7050-T7451 alloys as a function of applied potential
  - Establish what polarization levels would be desired for an MRP
- Explore the mechanistic underpinnings of the observed behaviors
- Use the data to explore the know variations in EAC behavior between alloy systems



#### Alloys of Interest: AA7075-T651 and 7050-T7451

#### Complex microstructure representative of material utilized in aging aircraft





#### From Corrosion of Aluminum and Aluminum Alloys (Davis)







#### Fracture mechanics-based method for assessing EAC behavior

### Utilize dcPD-based crack length measurements to enable K-controlled testing of specimens exposed to an applied potential while immersed in 0.6 M NaCl







#### Effect of applied potential on HEAC kinetics of AA7075-T6511







#### Can plot crack growth rate at iso-K to inform coating development



# **Observation 1:** There is a region of reduced susceptibility





#### Can plot crack growth rate at iso-K to inform coating development



**Observation 2: There is a loading rate dependence** (Suggestions a strain-controlled process, not a stress-controlled process)





#### Can plot crack growth rate at iso-K to inform coating development



# **Observation 3:** Cracking in the reduced susceptibility "well" is still very fast





#### The same analysis was performed for 7050-T7451







#### The same analysis was performed for 7050-T7451



# **Observation 1:** As expected, 7050-T7451 (Higher Cu, OA) cracks substantially slower than 7075-T651





#### The same analysis was performed for 7050-T7451



### **Observation 2:** 7050 exhibit reduced susceptibility "well" over the same potential range





- Systematically characterize the EAC behavior of 7075-T651 and 7050-T7451 alloys as a function of applied potential
  - Establish what polarization levels would be desired for an MRP
- Explore the mechanistic underpinnings of the observed behaviors
- Use the data to explore the know variations in EAC behavior between alloy systems



#### **Mechanistic Paradigm for Strain Controlled HEAC**







#### **Mechanistic Paradigm for Strain Controlled HEAC**















*Hypothesis:* This is due to a modification of the surface H content

This will be governed by the over-potential for H production at the crack tip

That overpotential will vary with applied polarization





### Experiments to quantify the extent of H generation as a function of potential (Scully)





















### 7075-T651

#### Critical Observation: - Potentials with reduced HEAC susceptibility corresponds with the reduced levels of H generation





### 7075-T651

#### Critical Observation:

 Potentials with reduced HEAC susceptibility corresponds with the reduced levels of H generation

#### Challenges:

- $H_2$  gas collection only a proxy for H-uptake
- H generation experiments not performed in crack tip solution
  - Sensitivity was analyzed
  - Sensitive at cathodic E where it goes alkaline
- H generation at anodic potentials (localized) versus more global for cathodic



- Systematically characterize the EAC behavior of 7075-T651 and 7050-T7451 alloys as a function of applied potential
  - Establish what polarization levels would be desired for an MRP
- Explore the mechanistic underpinnings of the observed behaviors
- Use the data to explore the know variations in EAC behavior between alloy systems



**Question:** Why is 7050-T7451 (High Cu, OA) less susceptible than 7075-T651













The similar trends observed for both systems suggests that the H generation is not dominating the differences between alloys







The similar trends observed for both systems suggests that the H generation is not dominating the differences between alloys







Closer examinations suggests that 7050-T7451 actually has modestly higher H generation at cathodic potentials in the transition regime



### 7075-T651





Closer examinations suggests that 7050-T7451 actually has modestly higher H generation at cathodic potentials in the transition regime



### 7075-T651







#### **Mechanistic Implications**



If Cu and OA is not impacting the chemistry that governs the surface H concentration (e.g  $C_s$ )...

## This then supports a governing role of:

#### - Intrinsic material response

- Impact of slip morphology
- Impact of H diffusion
- H redistribution
- Intrinsic failure criteria
  - Local defects
  - Solute segregation
- Sensitivity to the failure criteria to a given amount of H












The University of Manchester





## Understanding Performance of New Generation 7xxx Aluminium Alloys Prof Joseph Robson

joseph.robson@manchester.ac.uk DSTL/RAEng Chair in Alloys for Extreme Environments



**About Me** 

- Professor of Physical Metallurgy, University of Manchester UK
- DSTL/RAEng Chair in Alloys for Extreme Environments (2021-2026)
- Working on:
  - Metal fragmentation simulation and measurement
  - Modelling metal/laser interactions
  - Additive manufacture of AI alloys
  - EAC performance of new generation 7xxx AI
- Background
  - PhD in steel metallurgy (1996)
  - Industrial research fellow (British Aluminium/Alcoa) (1999-2003)
  - Academic in light alloy research group (2003-now)





#### EAC of New Gen 7xxx Alloys

- Major project at UoM (2017-2024) focused on understanding EAC performance of new gen 7xxx alloys for civil aerospace
  - Testing methods and crack growth measurements
  - Nano-scale microstructure studies
  - X-ray tomography studies of crack morphology
  - In-situ studies of crack initiation and growth
  - Modelling and simulation of initiation and growth



Phil Prangnell Academic lead



Tim Burnett Testing and tomography



Chris Race Atomistic simulation





**Pratheek Shanthraj** Phase field and crystal plasticity modelling

Henry Holroyd Consultant

 Now taking this understanding to apply to defence conditions and platforms (collaboration with UK DSTL and 1710 Naval Air Squadron)



#### **Development of 7xxx Thick Plate Aerospace Products**

The University of Manchester



- Thicker plate products (100 -280 mm) for machining integral components
- Higher strength and/ or same strength as 7050-T7651 with.....
  - o Lower quench sensitivity
  - Higher or equivalent damage tolerance corrosion performance



#### e.g. Alloys like 7449, 7055

 Increased Zn but maintain balance relative to MgZn<sub>2</sub>

Higher  $\eta' V_f \rightarrow higher$  strength



#### EAC in Humid Air (T76)

The University of Manchester

New gen 7xxx pass industry standard SCC testing (ASTM-G47) in saline solutions but...









Safety Information Bulletin Airworthiness SIB No.: 2018-04R1 Issued: 13 September 2018

Subject: Environmentally Assisted Cracking in certain Aluminium Alloys





#### **Static Load Tests - Lifetime**





## Influence of Humidity (SSRT)

The University of Manchester

- Slow strain rate tests at different humidity levels
- SR = 8 x 10<sup>-7</sup> and 5 x 10<sup>-8</sup>s<sup>-1</sup>, T = 70°C



Understanding the environmentally assisted cracking (EAC) initiation and propagation of new generation 7xxx alloys using slow strain rate testing

Y. Aboura<sup>a</sup>, A.J. Garner<sup>a</sup>, R. Euesden<sup>a</sup>, Z. Barrett<sup>b</sup>, C. Engel<sup>c</sup>, N.J.H. Holroyd<sup>a,d</sup>, P. B. Prangnell<sup>a</sup>, T.L. Burnett<sup>a</sup>,

partment of Materials, The University of Manchester, M13 9PL, UK bus: UK, Pepsias House, Aerospace Ave, Pilton, Bristol BSSA 7PA, UK bus, Albbas, Albbas Operations GmbH, Albbas-Allee 1, 28199 Bremen, Germäny symmer of Materials Science and Repubering. Gase Western Reserve University, Cleveland, OH, USA

#### Corrosion Science, 199, 1 May 2022, 110161



Sequence of EAC failure is now identified. Consistent with observations from in-service failure.

Focus on understanding initiation stages and transition to sustained crack growth



#### In-situ 4-point bend – Initiation 7085



MANCHESTER





 Trace back crack to unambiguously determine initiation point

R. T. Euesden et al. Corrosion Science 216 (2023) 111051





#### 7085 – Initiation Sites











## **Initiation Site Detail**

- Initiation always associated with pores/pore clusters
- Pores often correlated with intermetallic particles





#### **Examples of Initiation Sites**





#### **Typical Morphology – AA7085**

The University of Manchester



7085 70°C 50% 75%yield



#### **Crack Length Measurement**

The University of Manchester





V-K curves measured from individual cracks

Cracks are growing at K<<K<sub>1EAC</sub> determined from DCB test



The University of Manchester

# Sustained Crack Growth (DCB): Crack Path





#### **Effect of Recrystallized Grains**







#### Grain Boundary Microstructure and Chemistry





- No excess Zn at GBs in both alloys
- Difference in element segregation at GBs for 7050 vs 7085 quite small
  - 7050 higher Mg, lower Cu
  - 7085 lower Mg, higher Cu
- Does not directly explain H-EAC difference



The University of Manchester

#### **GB** Precipitates

- Cold fracture to expose GB precipitates
  - Identify 2 distinct GB ppt families
    - Quench induced
    - Ageing induced

AA7050 - 140 mm plate T/4







#### **GB** Precipitate Chemistry





## **Modelling and Simulation**

The University of Manchester

- Coupled phase field-crystal plasticity model for 7xxx EAC •
  - Hydrogen production
  - Migration to crack tip
  - Stress concentration and fracture





Atomistic modelling Work of separation of boundary with H concentration considering segregant elements



Simulating hydrogen-controlled crack growth kinetics in Al-alloys using a coupled chemo-mechanical phase-field damage model C. Grant, S. Roongta, T. L. Burnett, P. B. Prangnell, P. Shanthraj Preprint available on-line (under review Acta Mater.)



## **Summary: Critical Questions**

- How has your organization bounded the problem?
  - Range of different test types in humid air (static load, DCB, slow strain rate) + microstructural investigation
- What are the key influencing factors?
  - Temperature, humidity, steady-state stress, stress concentration...
- What are the most critical questions?
  - Complete mapping of behaviour of new-gen 7xxx in humid air as f(T, humidity, stress)...
  - Explore effects of complex environments (e.g. marine) and loading scenarios
  - Complete understanding of potential mitigating strategies (processing/temper/geometry)
  - Development of **predictive** physical models to be used as design tools
- UoM has capacity to address these questions and share results
- Challenges with translation: complex phenomena likely to require a combination of tests and distillation of complex models to simple rules
- Test methods (Henry Holroyd)
- New alloy development based on mechanistic understanding of the process + test methods sensitive to humid air EAC



The University of Manchester

#### **Thanks for listening!**

# EIC Testing of Commercial Al-Zn-Mg-Cu Alloys

#### Henry Holroyd<sup>1,2</sup> Tim Burnett<sup>1</sup> John Lewandowski<sup>2</sup> Matthew Curd<sup>1</sup> Geoff Scamans<sup>3,4</sup>

- 1 Department of Materials, The University of Manchester, M13 9PL, UK.
- 2 Department of Materials Science and Engineering, Case Western Reserve University, Cleveland, OH 44106, USA.
- 3 Innoval Technology, Banbury, Oxon OX16 1TQ, UK
- 4 BCAST, Brunel University, Uxbridge, Middlesex UB8 3PH, UK.

# What is needed to improve EIC Testing?

- Environmental test conditions relatable to 'Service Conditions'
- Initial alloy surface conditions relatable or representative of those expected during service use
- An understanding how local strain-accommodation and Primary Creep rates influence EIC initiation and early crack growth at mechanical-driving forces below K<sub>IEIC</sub> and Stress Thresholds.
- Clarification whether EIC 'crack-arrest' typically controls EIC initiation in structural applications

#### Constant-Load or Constant-Strain EIC Test



#### Instrumented Pseudo Constant Load Testing





# <u>Characterization of EIC Crack-Arrest Characteristics</u>

- Are EIC Crack-arrest characteristics at K's below K<sub>IEEC</sub> relatable to an Al-Zn-Mg-Cu alloys inherent EIC resistance?
- If Yes, we can develop a 4-point bend EIC Test Method to characterize an alloys inherent EIC resistance under experimental conditions relatable to those for a commercial structural application.
- Success will require the use of appropriate initial surface condition for EIC initiation along with environmental and mechanical loading conditions relatable to expected service conditions





EIC Crack Length



# New Approach to evaluate EIC propensity

- Determine if EIC Crack-arrest behavior at K's below K<sub>IEIC</sub> is related to an Al-Zn-Mg-Cu alloys inherent EIC resistance.
- Quantify the role local 'strain-accommodation' plays during EIC initiation and early-stage crack growth

Metal/environment systems where exhaustion of primary creep ahead of exposure of a stressed sample to an EIC environment can either prevent or significantly increase minimum mechanical driving force ( $G_{Thresshold}$  or  $K_{IEIC}$ ) required for EIC initiation.

Alloy	Loading Conditions	Environment Conditions	Temperature	T <sub>m</sub> (T/T <sub>mp</sub> ) °K	Reference
Ti-7Al-2Cb-1Ta Annealed YS = 706 MPa, UTS = 823 MPa, K <sub>IC</sub> = 121 MNm <sup>-</sup> <sup>3/2</sup>	Fatigue Pre- cracked SEN cantilever bend, Constant Load	Lab Air (30% RH) or 0.53M NaCl, FCP	RT	0.15	Leckie 1967
Mg-7AI	Constant Load Smooth Tensile Specimen	Aq. CrO4²-/NaCl	RT	0.34	Wearmouth et al. 1973
26Cr-1Mo Ferritic Stainless Steel	Constant Load Smooth Tensile Specimen	42% LiCl + Thiourea -500 or -460 mV (SCE) De-aerated	140 <sup>o</sup> C	0.25	Kwon et al. 1992
Annealed 304 Stainless Steel K <sub>IC</sub> = 86 MNm <sup>-3/2</sup>	Constant strain Notched WOL Constant Load SEN both with 0.2 mm notch root radius	42% MgCl <sub>2</sub>	143 <sup>o</sup> C	0.25	Gu et al. 1994
As-quenched NiCrMoV Rotor Steel, YS=1250 MPa	Double Cantilever Beam, Fatigue Pre-cracked Constant Load & Constant Strain	Dry Air (Silica–gel) Distilled Water	22 °C	0.17	Rieck et al. 1989
As-quenched 4340 high strength martensitic steel, YS 1700 MPa	Double Cantilever Beam, Fatigue Pre-cracked Constant Load & Constant Strain	Dry Air (Silica–gel or P <sub>2</sub> O <sub>5</sub> ) Distilled Water	22 <sup>0</sup> C	0.17	Rieck et al. 1989
C-Mn Pipeline Steel	Constant Load Fatigue Pre- cracked SEN cantilever bend,	1M Na <sub>2</sub> CO <sub>3</sub> + 1M NaHCO <sub>3</sub> -900 or -650 mV (SCE)	75 <sup>o</sup> C	0.2	Parkins 1979

Holroyd et al (2024)




## Measurement of Environmentally Assisted Cracking in Atmospheric Tests

Fritz Friedersdorf Carly Cocke

AFRL-FAA Technical Interchange Meeting (TIM) on Environmentally Assisted Cracking (EAC) / Stress Corrosion Cracking (SCC) of High-Strength 7000 Series Aluminum Alloys

5 November 2024

## Applied Research and Development

Continuous *in situ* monitoring and mechanical testing of aluminum alloys in corrosive atmospheres

- Relevance of accelerated laboratory test to outdoor exposure
- Significance of galvanic couples and crevice conditions
- Risk of chromate elimination from protective coatings for EAC and ASIP assumptions

Combined-effects laboratory and relevant environment testing

- Environmental conditions
  - RH, temperature, and conductance (salt loading)
- Compliance measurements used to estimate crack growth







### Measurement Sample and Test System

Fracture sample designed to accommodate range of alloy orientations, crevice former, and surface finishes

• No need for a precrack

Mechanical test system for continuous measurement of cracking throughout a combined effects test



## Influence of RH and Salt Chemistry

High transient crack growth rates for AA7075-T651 occur preferentially during drying (decreasing RH)

Hygroscopic properties of the salts influence RH dependence

Results consistent with testing that emphasizes cyclic wetting and drying (ASTM G47)









## **Galvanic Crevice**

Static loading tests using a statistical representation of a RH diurnal cycle

Measure galvanic current (aluminum and SS316) and crack growth rate (CGR)

Galvanic corrosion rate peaks during both drying and wetting

• ORR dependent on thin film conditions

CGR peaks when humidity decreases (drying), and cracking is relatively insensitive to wetting

• Drying focuses corrosion into the crevice/crack





## Mechanistic Interpretation of Atmospheric EAC

Variable chemistry, reaction rates, and spatial distribution of electrolytes

- High salt concentrations
- Increased ORR for thin films
- Current distribution on surfaces and crevices







BEASY doing 3-D modeling of fracture sample and current distribution for specific environmental conditions



## Mechanistic Interpretation of Atmospheric EAC

During drying electrolytes and corrosion currents are localized to crevices and cracks (last areas to dry off)

- ORR at mouth
- Anodic dissolution near the ORR mouth area
  - Hydrolysis lowers pH and promotes hydrogen ion reduction
- At the crack tip, hydrogen ion reduction may be favored
  - Proton reduction may increase pH at crack tip

#### Consistent with HEAC described by Gangloff, Cooper, and Kelly



 $O_2 + H_2O + 4e^- \rightarrow 4OH^-$ 

 $M \rightarrow M^{n+} + ne^{-}$ 

$$M^{n+} + zH_2O \rightarrow M(OH)_z^{(n-z)+} + zH^2$$

$$H^+ + e^- \rightarrow H_{ads}$$



## **Protective Properties of Coatings**

- Susceptible aluminum alloy facture samples have been used to evaluate the EAC protective properties of coatings
- Working with NAWCAD to refine method and update AMPP TM21449-2021
- Need to verify results in outdoor exposure testing

AA7075-T651 MDACT Applied load of 30% YS (140 MPa, 325 N) S-L crack orientation













## Dynamic EAC - Load Frame with Actuation

Dynamic EAC system for testing in laboratory and outdoor environments DEAC load frame:

- Force Range:  $0 1000 \text{ N} [223 \text{ Ib}_f]$
- R-ratio: 0 to 1
- Frequency: constant load to 5 Hz sine wave

For reference: 1000 N produces 90% YS on smooth gauge AA7075-T651 DCB sample





## Moving Forward

Conducting outdoor EAC testing at Battelle FMRF to verify RH and load dependencies in relevant environments

- Combined with environment, contaminant, and corrosivity measurements for modeling corrosion and cracking
- Perform DOE with a wide range of coatings (Cr and non-Cr) in collaboration with NAWCAD using MDACT
  - Results will be used to revise AMPP TM21449-2021
  - Validate coating performance results in outdoor tests

Initiate corrosion fatigue testing using similar conditions as SCC work (RH, salt, crack orientation, galvanic coupling)

• Dynamic environmental and mechanical processes





## Fritz Friedersdorf https://acuitycorrosion.com/ fritz.friedersdorf@lunalabs.us +1 540 808 8947

## **AFRL-FAA EAC TIM**

## SOUTHWEST RESEARCH INSTITUTE®

### 11/5/2024





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## Several ways to think of EAC on aircraft

- Historically
  - Effects of environment were accounted for in model uncertainty
  - This has worked well and improved with experience
- Concerns arose because of
  - Aloha flight 243
  - Chromate elimination
- How do we better account for corrosion in structural integrity?
  - Affect of damage profile on subsequent fatigue (sequential approach)
  - Affect of environment directly on CGR (parallel approach)







MATERIALS ENGINEERING

2



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# on crack growth?



and temps Higher RH

## Effects of RH don't look minor when viewed as $da/dN vs \Delta K$ .





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#### **MECHANICAL ENGINEERING**

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## **Cyclic wetting and drying affect fatigue**



CGR increases by 2.5x – 5x upon drying







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MECHANICAL ENGINEERING

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## Under what conditions might we see SCC of **AA7075-T6**?

- Constant K from 0 to 16.49 MPa $\sqrt{m}$  and immersed in deaerated sea water
- For K > 0, CGR initiates around -0.71 V (Ag/AgCl)
- CGR can continue to lower values but generally ceases as the potential falls below -0.75V (Ag/AgCl)
- Implications
  - Cracking possibly initiated under aerated OCP conditions (OCP at the breakdown potential  $(E_{bd})$









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## **Galvanic interactions likely increase CGR**



- Galvanic current drives CGR
- CGR = dissolution controlled









#### MATERIALS **ENGINEERING**

7

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## An aggressive crack tip chemistry can lead to sustained CGR well below -0.71 V(Ag/AgCI)

### Scan

- Original OCP = -0.745 V(Ag/AgCI)
- 12 cyclic scans of OCP to -0.65 V(Ag/AgCI)
- Potential released to OCP
- OCP immediately falls to -0.92V(Ag/AgCl), consistent with low pH values and high  $AICI_3$
- CG observed below critical potential range and until OCP begins to recover to initial value
- Implications
  - Cracking occurs until the environment at the crack tip is diluted by diffusion or exhauasted









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## Under atmospheric drying, the observed beavior is different than in solution.

- Cycle: Initiation at constant current density under immersion. Drain solution and maintain at 90% RH. Blow in dry lab air.
- As RH falls below 75% RH, large jump in CGR
- IG cracking observed with minimal corrosion
- Hypotheses
  - Increased H uptake relative to outward diffusion
  - Moisture dries from broad IGC sites but remains in GBs (very sharp crack)
  - Combo of both







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## What is SwRI doing?

- Working with ASIP community with interest in defining a test method
- Early goals
  - Can the cyclic exposure be used to compare SCC performance of AA7xxx alloys
    - Tighten up some experimental details
    - Optimize the cycle to highlight material performance differences
  - Expected funding starting Q2 FY25 to look at 3 to 4 alloys
    - 3 weapons platforms
    - In discussions with I OEM
- Mid-term goals
  - Using the developed cycle, move from alloy testing towards system testing, including protection systems and structural components





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SAFE



## **Prior Work**

- SAFE and OSU have worked together, separately and with various other partners on improving testing methods for EAC of 7xxx series aluminum alloys under atmospheric conditions to better predict this type of damage
  - Work under the Technical Corrosion Collaboration (FA7000-18-2-0001) examined effects of salt loading and other environmental effects on AA7085-T7451
  - This was leveraged by SAFE on work refining the developed environmental control system for this type of testing which has the ability to combine salt films, RH, salt spray, various background gases and temperature (FA7000-19-2-0026)
  - Work has highlighted to need to understand the drying cycle on the increased presence of environmental cracking on newer 7xxx series alloys





**Chamber Design** 

Environmental variables include:

**Relative Humidity** 10%-100% Ozone 30 ppb-100 ppm Salt 10-1000 μg/cm<sup>2</sup> UV-light UV-A Temperature -60-150 F





\*Considerations for Laboratory Corrosion Fatigue Testing of Aluminum Alloys for DoD Applications. Galyon Dorman, Rausch, Niebuhr - 2023 DoD Corrosion Conference



#### **Continued Refinement with Chamber Design**

- Thin film for simulating atmospheric corrosion NaCl Solution of NaCl evaporated onto masked specimen
- Desire even dispersion of NaCl on sample surface
- Complex system requires refinement whenever a new environmental parameter is added
- Can be used on any sample design







### COLLEGE OF ENGINEERING

1.5 in

\*Considerations for Laboratory Corrosion Fatigue Testing of Aluminum Alloys for DoD Applications. Galyon Dorman, Rausch, Niebuhr - 2023 DoD Corrosion Conference

5.55 in

AA7085-T7451

## Some variables aren't as impactful as others: $O_3$ and salt load density above $^{1000 \text{ug/cm}^2 \text{vs} 400 \text{ug/cm}^2 \text{vs} 50 \text{ug/cm}^2 \text{printed salt,}}$ $^{2}100 \,\mu\text{g/cm}^2$



- > The presence of ozone continues to have limited effect on the crack growth rates.
- > Salt loading variation between 50-1000  $\mu$ g/cm<sup>2</sup> does not appear to change crack growth rates.
- > Salt films with lower concentrations of salt can be more suspectable to unexpected drying
- > Currently there is limited difference noted between samples printed with salt and those with pipetted salt.

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\*Considerations for Laboratory Corrosion Fatigue Testing of Aluminum Alloys for DoD Applications. Galyon Dorman, Rausch, Niebuhr - 2023 DoD Corrosion Conference



#### Crack growth rate during drying is dependent on drying rate



## Fractography is consistent with previous findings of a transition from transgranular to intergranular cracking



### At low f (< ~1 Hz), all Cl<sup>-</sup> containing environments have = da/dN At higher f, atmospheric has accelerated da/dN



- ➤ 300 µg/cm<sup>2</sup> NaCl on surface and 80% RH ↑ da/dN over air with no salt
- 1 Hz and below, full immersion approximates atmospheric da/dN
- Above 1 Hz, atmospheric da/dN higher than similar salinity full immersion
- Approaching 10 Hz and higher, atmospheric da/dN is slightly higher than 0.06M NaCl
- conventional full immersion
  environments are suitable at lower *f*,
  but may underestimate at higher *f*



## A Current Program is Funded By ONR:

- Develop a Digital Twin to advance the state of the art for structural integrity modeling specifically with regards to corrosion and the transition to corrosion fatigue
  - Focus will be on AA7075-T6 for model development to ensure success, particularly with regards to large historical datasets available
  - 1) Data mine and pull in data from other programs on corrosion and corrosion fatigue behavior as a function of atmospheric environmental variables (RH, T, O<sub>3</sub>, UV, salt loading) to begin to build data driven Bayesian approach
  - 2) Experimental examination of the effects of atmospheric environmental variables on corrosion fatigue crack growth rates on alloy itself where literature data are critically lacking
  - 3) Perform sheltered outdoor tests to obtain validation data for digital twin
  - 4) After initial creation, begin to incorporate the effect of bolts and coatings by on both corrosion and corrosion fatigue by integrating data from other similar work





### A Current OSU Program Funded by NSF

Hypothesis:

2xxx Al alloys have an intrinsic resistance to EAC compared to 7xxx alloys because Cu containing precipitates within the crack wake dealloy causing Cu to replate and accelerate cathodic reaction kinetics in the crack environment facilitating a more basic environment where a passive film can be stabilized

**Objectives:** 

- <u>Actively probe crack tip pH in 2xxx and 7xxx Al alloys to gain evidence in support of the</u> <u>fact that 2xxx have pH within range for Al passivity when da/dN is low</u>
- Cast experimental compositions to varying the presence of dealloying/Cu replating precipitates
- Investigate effects of frequency on da/dN on experimental compositions
- Electrochemical characterization of compositions to determine propensity to passivate/repassivate



### AA7075-T6 shows acidic crack under OCP



- 7075-T6 shows an acidic crack as low as 3.5 at 0.1 Hz
- @ 1 Hz mostly between 5.4 and 7

Montiel, Marino, Papageorge, Locke. Manuscript in progress.



## **Next Steps**

- Confirming the conditions that consistently reproduce the damage in laboratory testing
  - Suspected wetting and drying conditions along with stress and temperature
- Determine the microstructure features that are driving the issue
- Develop a repeatable test methodology to screen for this issue in alloy development going forward
  - Consider that method may need to be somewhat flexible to allow for other unknown conditions of risk to be studied later if next generation alloys also show issues
- Because most of SAFE and OSU's work on this topic has been funded by governmental organizations the work should be sharable and available for



validation.

**5-6 NOVEMBER 2024** 

### CMI BEASY SOFTWARE TOOL DEVELOPMENT & CAPABILITY OVERVIEW

AFRL-FAA TECHNICAL INTERCHANGE MEETING (TIM) ON ENVIRONMENTALLY ASSISTED CRACKING (EAC) / STRESS CORROSION CRACKING (SCC) OF HIGH-STRENGTH 7000 SERIES ALUMINUM ALLOYS



### Company Overview

- Developer of advanced modeling and simulation • tools for corrosion and fracture mechanics analysis
- Modeling services to support design requirements, • asset management, and sustainment planning
- Extensive R&D work guides creation of client-driven, • custom software solutions





FΔ





**PROVIDING DURABILITY AND** STRUCTURAL INTEGRITY SOLUTIONS WORLWIDE



### Corrosion & Fracture Mechanics Modeling Capability

- Fracture Mechanics:
  - BEM driven FCG simulations.
  - Automated remeshing and crack growth.
  - LEFM (residual stress, multi-site dam age)
  - New advanced DDM solver
- Corrosion Manager:
  - FEA simulation package for galvanic corrosion for thin film and bulk electrolytes
  - Calculates IR drop through electrolyte for realistic component geometries.
- PolCurveX:
  - Materials database with tools for theoretical analysis of polarisation curves and deconvolution (MIL-STD-889).
  - 1d curve crossing analysis
- Actively engaged in collaborations to expand and combine these into toolkits.




## Evolving EAC Modeling Framework

- Proven simulation methodology supporting:
  - Current density and potential distribution prediction for galvanic couples in both thin film and bulk electrolytes.
  - SIF solutions (mixed mode) for realistic models and crack surface geometry
- Capabilities include:
  - Empirical crack growth rate equations
    - Arrhenius equation, others to be included
  - Tabular representation of crack growth rate as a function of environment and applied stress:
    - da/dt vs K data sets for different environmental conditions.



Stage I - K dependence

03894337

0997109

89552746

79343575

.69134403

22192619

.48716059

Stage II - Temperature and corrosive environment main driver, crack growth rate independent of K



BEASY Crack Growth Wiza

......



## More realistic modeling of crack surface loading (conceptual)

Can we introduce  $H_2$  charging effects?



De-convolution	analysis	[MIL-STD-889]:
AA7075 in 3.5%	NaCl e le o	ctrolyte

Hydrogen	Evolution	
Sym.	Value	Unit
b <sub>H2</sub>	0.11	Vdecade
E <sub>H2</sub>	-0.411	V
$i_0^{H2}$	1.62E-10	A/cm2
i <sub>lim</sub> H2	0.01122	A/cm2
$\begin{array}{lll} b_k & : \text{ Tafe} \\ E_k & : \text{ Equ} \\ i_0^k & : \text{ Excl} \\ i_{LIM}^k & : \text{ Lim} \end{array}$	l slope, i.e. $b_k =$ ilibrium potentia nange current de iting current der	= 2.3 <i>RT/(anF)</i> al ensity usity



## Collaboration with Luna Labs - SCC Modeling

- Collaborative effort with Luna Labs to:
  - Assist in calibration of Luna's SLEAC measurement system (compliance based) by determining SIF solutions for range of crack sizes at given load



• Integrate lab/field measured SCC crack growth rates into an environment-driven fracture mechanics framework that can be used as a predictive tool for realistic component geometry (ongoing)



Double Cantilever Beam (DCB) is subject to sustained loading, under varying environmental conditions. A crevice former accelerates crack initiation in the notch area



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- Build atmospheric corrosion models for different stages of wetting/drying cycle (BEASY Corrosion Manager)
- Use BEASY Digital Twin methodology and Luna Acuity sensor dynamic measurement data to determine accurate model in put parameters
- In vestigate relationships between drying cycles and peak crack growth rates. Develop methodology to incorporate in a EAC modelling framework for different operational environments (ongoing)





- Leverage knowledge to improve/expand modeling methodology (e.g. empirical SCC equations specific for 7XXX series alloys)
- Leverage new test data to populate and drive models. Access representative input parameters (e.g.  $E_A$ ,  $K_{Iscc}$ , da/dN for insolution environments)
- Academ ic research that may improve development of computational methods (e.g. consider H<sub>2</sub> charging or dissolution mechanisms in crack advancement algorithms)
- Continue to iterate and build out new capability based on feedback from EAC research community with a goal of having engineering level tools that can be used in practice





## THANK YOU

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