# Microstructure and Mechanical Properties
# Evolution of Long-time Serviced 5xxx Aluminum Alloys


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## ABSTRACT

Al-Mg (5xxx) alloys are commonly employed in storage tank, pressure vessel, and marine service applications that require a weldable and moderate-strength alloy with good corrosion resistance. However, experience has shown that 5xxx alloys become susceptible to intergranular attack, intergranular stress corrosion cracking, and reduced ductility and toughness when exposed to temperatures ranging from 50°C to 200°C for sufficiently long periods of time. This phenomenon is known as "sensitization". It had been theorized that the sensitization phenomenon may be caused by the formation of β-phase precipitates on grain boundaries during the elevated-temperature exposure. In order to investigate the change of mechanical properties of Al-Mg alloys after long-time service, commercially available 5083-H116 and 5456-H116 alloys have been sensitized up to 1000 hrs at 80°C, 100°C, and 175°C. Room temperature tension, fracture toughness, fatigue crack growth and hardness tests have been completed to determine changes in mechanical properties resulting from the sensitization. Additionally, similar mechanical testing has been run on long-term serviced material (i.e. after 15 years service). Metallographic observations have been used to investigate the grain size and the distribution of secondary phases, while TEM/STEM have been used to analyze precipitates in the grains and/or on grain boundaries. TEM observations of long-term serviced material show no obvious evidences that β-precipitates were formed during the service. Additionally, the mechanical properties of the long-term serviced material do not exhibit significant changes. However, investigation will continue by heat treating additional 5083-H116 and 5456-H116 specimens for extended times at intermediate temperatures and using mechanical testing to investigate the presence and/or effect of β-phase precipitation.


## EXPERIMENTAL

### Materials

- Commercial 5456 (H116) and 5083 (H116) aluminum alloys
- Commercial 5456 aluminum alloy removed from service after a 15-year exposure

### Microstructure

- Metallography
  - AA 5456 etched by Keller's Reagent
  - AA 5083 etched by a modified Poulton's Reagent
- TEM/EDS
  - Samples prepared by mechanical polishing and ion milling
  - Observation and analysis conducted on Philips CM20 at 200kV
- STEM/EDS
  - Observation and analysis conducted on FEI Tecnai F30 at 300kV

### Mechanical Testing

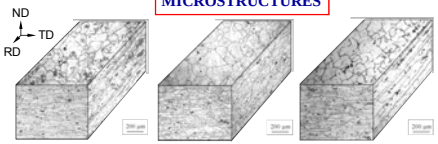
- Hardness: Rockwell hardness tester ( 1/16" ball, 100Kg).
- Tension Test: dog bone sample, displacement rate of 0.05mm/min, Instron testing machine.
- Notched toughness, fatigue precracked toughness, fatigue crack growth measured.
- Toughness and fatigue crack growth tests:3PB specimen: 200 μm notch root radius.
- MTS 20 Kip closed loop servohydraulic machine, MTS 458.20 controller, FTA control software.
- Specimens fatigued at 20 Hz, sinusoidal wave, load ratio (R) =0.1.
- Fatigue crack length measured with metallic foil KRAK© (KG-A05)-gages monitored by a Fractomat model 1288 crack measurement system.

### Heat Treatment

- Blue M Furnaces
- Temperatures: 80°C, 100°C, 175°C
- Times: 1 hr, 10 hr, 100 hr, 1000 hr
- Future Study Times: 2000 hr, 5000 hr, 10000 hr

## RESULTS

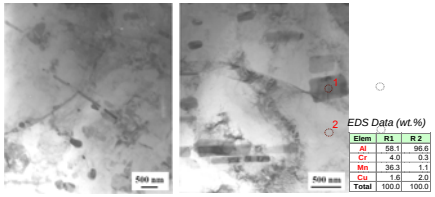
### MICROSTRUCTURES




*Metallographic pictures showing grain boundaries in three dimensions.*

*Grain Size Measurement (μm)*

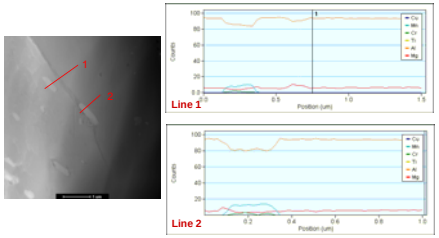
| Sample | Direction | ND Plane | TD Plane | RD Plane |
|---|---|---|---|---|
| 5456(H116) | RD | 20.3 ± 4.2 | 30.7 ± 4.7 | |
| | TD | 15.1 ± 2.1 | | 25.3 ± 3.3 |
| | ND | | 11.5 ± 0.8 | 11.7 ± 1.2 |
| | Aspect Ratio | 1.1 | 2.7 | 2.2 |
| 5456(Sensitized) | RD | 18.0 ± 3.7 | 16.8 ± 3.4 | |
| | TD | 17.6 ± 2.3 | | 18.3 ± 2.9 |
| | ND | | 8.1 ± 1.2 | 8.3 ± 0.9 |
| | Aspect Ratio | 1 | 2.1 | 2.2 |
| 5083(H116) | RD | 18.3 ± 1.9 | 35.0 ± 8.0 | |
| | TD | 13.8 ± 2.0 | | 30.6 ± 9.9 |
| | ND | | 9.3 ± 1.2 | 10.2 ± 0.9 |
| | Aspect Ratio | 1.3 | 3.7 | 3.0 |




*EDS Data (wt.%)*

| Elem | R1 | R 2 |
|---|---|---|
| Al | 58.1 | 96.6 |
| Cr | 4.0 | 0.3 |
| Mn | 36.3 | 1.1 |
| Cu | 1.6 | 2.0 |
| Total | 100.0 | 100.0 |

*TEM views showing precipitation at grain boundaries and in grains. Particles are rich in Mn and Cr, analyzed via EDS.*



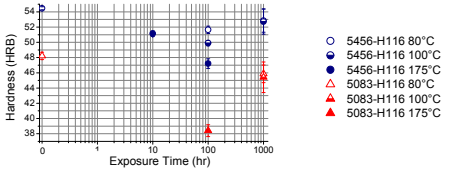

*STEM view showing precipitation at grain boundary and in grain. Corresponding EDS line profiles showing particles containing much more Mn and grain boundary containing more Mg than matrix, respectively.*

## RESULTS

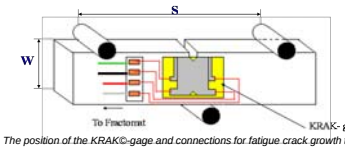
### HARDNESS-TENSION

| Materials | HRB | $\sigma_y$ (MPam$^{1/2}$) | UTS (MPam$^{1/2}$) | EL % |
|---|---|---|---|---|
| 5456(H116) | 52.3 ± 0.6 | 255 | 357 | 17.2 |
| 5456(Sensitized) | 36.5 ± 1.0 | N/A | N/A | N/A |
| 5083(H116) | 44.5 ± 4.5 | 265 | 340 | 15.3 |

### THERMAL EXPOSURE EFFECT

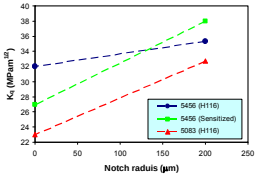



### FRACTURE TOUGHNESS

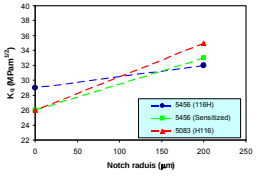



*The position of the KRAK©-gage and connections for fatigue crack growth test.*

### Longitudinal Direction




### Transverse Direction




- ***Sensitized 5456 exhibits reduced $K_q$ and increased Paris slope.***


## ACKNOWLEDGEMENTS

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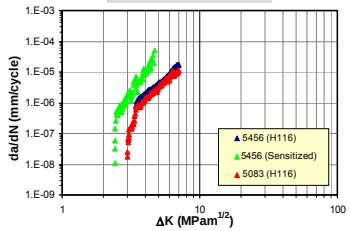
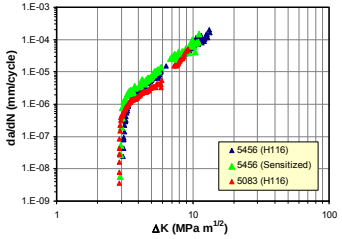
## RESULTS

### FATIGUE CRACK GROWTH

#### Longitudinal Direction




#### Transverse Direction




### FATIGUE CRACK GROWTH SUMMARY

| ID | Testing Direction | $\Delta K_{th}$ (MPam$^{1/2}$) | m | $K_q$ (MPam$^{1/2}$) |
|---|---|---|---|---|
| 5456(H116) | Longitudnal | 3.0 | 3.6 | 32 |
| 5456(Sensitized) | Longitudnal | 2.4 | 6.3 | 27 |
| 5083(H116) | Longitudnal | 3.0 | 3.8 | 23 |
| 5456(H116) | Transverse | 3.1 | 3.2 | 29 |
| 5456(Sensitized) | Transverse | 2.9 | 3.7 | 26 |
| 5083(H116) | Transverse | 2.9 | 3.0 | 26 |

## CONCLUSIONS

- Microstructures of as-received and sensitized materials quantified via metallography and TEM.
- Preliminary fracture toughness, fatigue crack growth , and hardness behavior characterized.
- Future work to examine effects of thermal exposure on mechanical properties.