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An Analysis of Variability in Sensitization Behavior of 5083-H131 and the Impacts on Yield Strength

by Heather Murdoch, Daniel Magagnosc, Lindsey Blohm, Denise Yin, and Tim Walter

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14. ABSTRACT Multiple plates of 5083-H131, representing a variety of suppliers and plate thickness, were assessed for their susceptibility to sensitization. More variability in the degree of sensitization was observed at the lower sensitization temperature (70°C). The H131 behavior was also compared to an example 5083-H116 plate that was known to be highly susceptible to sensitization. All sensitized plates and conditions exhibited a decrease in the yield strength. Microstructural characterization was performed on one plate in conjunction with a detailed examination of strengthening model parameters to establish the specific mechanisms contributing to the yield drop. The yield drop in sensitized 5083 is caused by Mg leaving solid solution to form the β phase and dislocation recovery. The proportional effect of these two mechanisms will vary depending on plate (e.g., its initial dislocation density, grain boundary density and character, initial precipitate density, and overall Mg content) but it is generally expected to be influenced more by the dislocation recovery.					
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1. Introduction and Motivation

The 5xxx series of aluminum alloys is Al-Mg based and is used on multiple Army and Navy platforms due to its good strength, general corrosion resistance, and weldability. The material specifications for both services stipulate a set of minimum acceptance mechanical properties and corrosion behavior to be met in order to be qualified for use. The minimum yield strength requirements for H116¹ and H131,² for example, are shown in Table 1.

Table 1 Minimum mechanical properties of 5083 by thickness^{1,2}

Thickness (inches)	Yield strength, 0.2% offset (MPa) H116 / H131	Tensile strength (MPa) H116 / H131	Elongation (%) H116 / H131
0.063 to 0.249	214 / NA	303 / NA	10 / NA
0.250 to 0.499	214 / 241	303 / 310	10 / 8
0.500 to 1.250	214 / 255	303 / 310	12 / 8
1.251 to 1.500	214 / 255	303 / 310	12 / 8
1.501 to 2.000	200 / 255	283 / 310	12 / 8
2.001 to 3.000	200 / 241	283 / 303	12 / 9
3.001 to 10.500	NA / 214	NA / 296	NA / 11

Note: NA = not applicable.

The yield strength in 5xxx alloys is predominantly derived from a combination of the Mg content in solid solution and dislocation structures produced via cold work.

In the case of 5xxx alloys, the supersaturated solid solution has a corrosion-related side effect called sensitization. As the Mg is supersaturated in solution (i.e., present in concentrations higher than the equilibrium solubility limit), under certain conditions (e.g., sufficient temperature and time) the Mg will leave solid solution and form the equilibrium β phase (Al_3Mg_2). The β phase is anodic with respect to the solid solution cathodic matrix and will preferentially corrode. Of particular concern to in-service behavior is that the β phase predominantly forms on grain boundaries (heterogenous nucleation) where corrosion of the β phase leads to intergranular corrosion cracking and subsequently can result in stress corrosion cracking.

The majority of sensitization research focuses on the impact of β phase formation on the corrosion behavior; however, as the formation of the β phase is a loss of Mg in solid solution, sensitization will also impact the yield strength. This was shown in prior work,³ where a sample of 5083-H131 exhibited a 60-MPa drop in the yield strength at only an intermediate level of sensitization.

Sensitization progresses as a result of exposure to temperatures of approximately ≥ 40 °C. Numerous studies have measured the increase in the Degree of Sensitization (DoS) over time at a range of temperatures. An analysis of literature data sets across four 5xxx alloys and five tempers⁴ highlighted the variability of sensitization behavior. Even within the same alloy and temper, significant variation occurs, as shown in Fig. 1 for data sets of the same alloy and temper designation sensitized at the same exposure temperature. The variation continues to persist even at long exposure times (some data sets have measurements past 700 days exposure); the lines in Fig. 1 are fits to the data using a kinetic model.⁴⁻⁶ Considering the DoS value of 25 mg/cm² (threshold for the material to be considered sensitized per the ASTM),⁷ Fig. 1 shows that a given component of 5083-H116 could sensitize anywhere in a range of 1 day to approximately 75 days when at an exposure temperature of 100 °C.

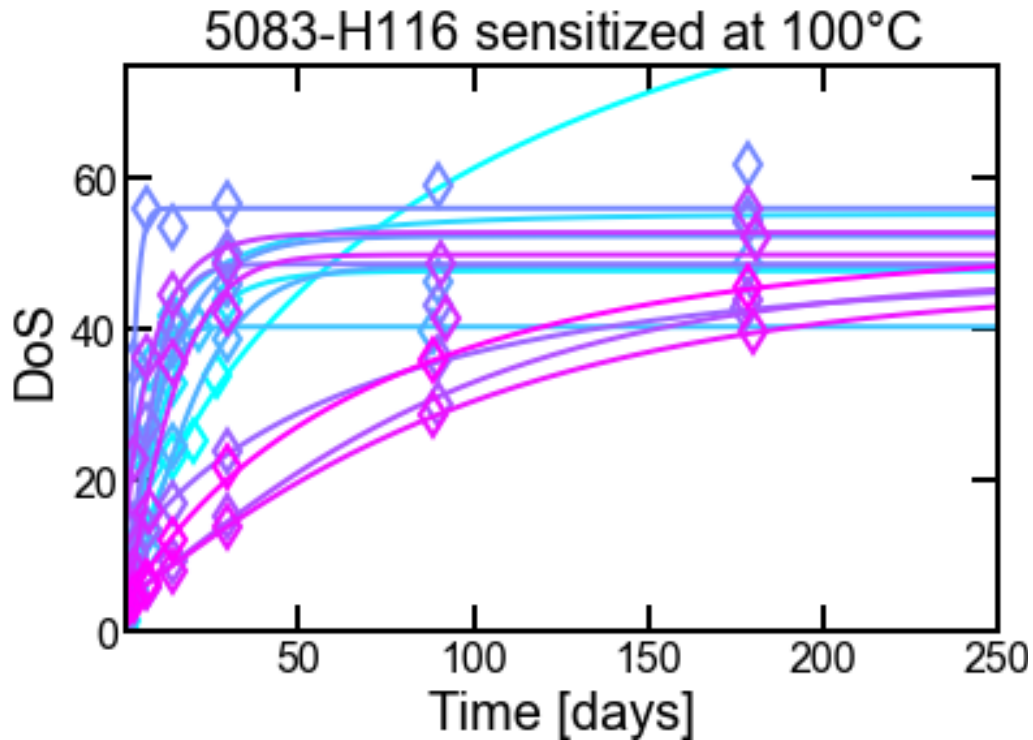


Fig. 1 Examples of data sets for 5083-H116 sensitized at 100 °C. Points are experimental measurements and lines are fits to the data.

It is expected that sensitization will vary between alloys and tempers based on the mechanisms of β phase formation. Broadly speaking, increased Mg content in the alloy would lead to larger amounts of β phase, while tempers with more cold work provide more kinetic pathways (via dislocation density) for the Mg to accumulate and form β . The saturation of the DoS (Fig. 1) in a material is related to the total amount of β that can form, and the kinetics of formation are reflected in the slope.

The nucleation sites for β formation would contribute to both the total amount and the kinetics, where an increase in the density of nucleation sites (i.e., grain boundaries and pre-existing precipitates) could increase the amount of β formed while the spacing between them could reduce the distance for Mg to diffuse, enhancing the kinetics. Here we discuss these main factors anticipated to affect sensitization in 5xxx alloys with respect to the microstructural features of dislocations, grain size, precipitate phases, and solid solution solute content. These features are also those that contribute to the yield strength in aluminum alloys; therefore, we expect the yield strength of sensitized material to vary with respect to its sensitization condition. There are quantified relationships governing the impact of microstructure on yield strength, but only qualitative descriptions linking the features to sensitization.

1.1 Overview of the Microstructure

The typical microstructure of a 5xxx alloy and the corresponding strengthening mechanisms are depicted in Fig. 2; the height of each color bar is indicative of their relative contribution to the total yield strength as calculated here for an example plate of 5083-H131 where dislocation strengthening is the largest component.⁸ 5xxx alloys are cold worked (deformed via rolling, stretching, etc.) to increase the dislocation density. Dislocation content is highly challenging to accurately quantify. A variety of diffraction and electron microscopy methods have been devised;^{9–12} however, each method suffers from several drawbacks. For instance, dislocation tangles or substructures can be observed with high resolution microscopy, as in the lower central image of Fig. 2 taken from a plate in this study. In this case the visible dislocations represent a partial assessment of the dislocation content as the specimen is necessarily thin to ensure electron transparency, visibility depends on the local zone axis, and strain fields introduce significant additional contrast thereby masking some dislocation line segments.

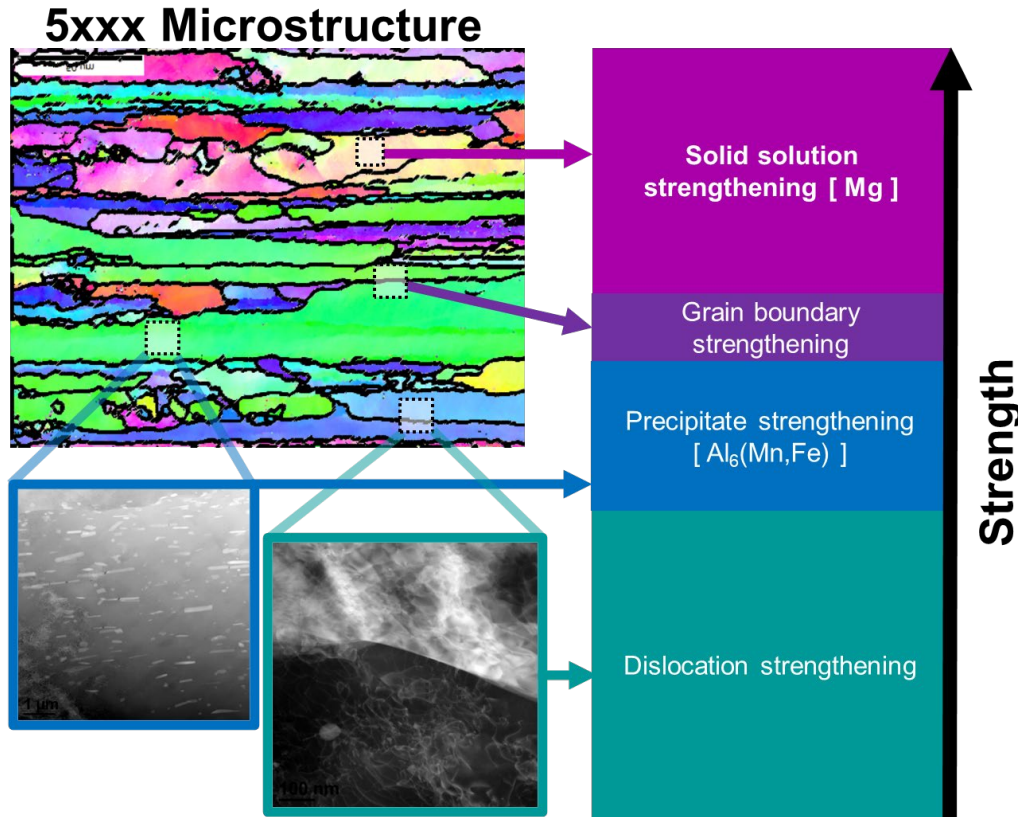


Fig. 2 Schematic of 5xxx microstructure features at varying length scales and their associated strengthening mechanisms. The microstructural images are from a plate of 5083-H131 examined in this study.

The grain structure in the EBSD image of Fig. 2, upper left, is indicative of a cold worked aluminum plate, with large, elongated grains. Smaller, more equiaxed grains can also form via recrystallization in order to minimize the free energy after sufficient dislocation density is introduced and when sufficient thermal energy is available to increase dislocation mobility.

Both large particles (10–100 μm) and small precipitates (100–500 nm) of the Al₆Mn phase are generally present in 5xxx series alloys; other large particle phases may be present (e.g., Mg₂Si) depending on the exact composition of the alloy and the contamination during casting. An example of the small-scale Al₆Mn particles is in the lower left inset of Fig. 2. The β phase is not typically expected to be present in the initial microstructure in large quantities. It has occasionally been observed in extremely high deformation experimental processes.^{13,14} Such processes are atypical of those employed in commercial plates; consequently, preexisting β phase is discounted.

1.2 Impact of Microstructural Features

The attendant impact of the microstructural features on both sensitization and yield strength is briefly summarized in Table 2. The yield strength can be estimated by a linear combination of contributions from different strengthening mechanisms:

$$\sigma_{yield} = \Delta\sigma_{grain} + \Delta\sigma_{ss} + \Delta\sigma_{DW} + \Delta\sigma_{precip} \quad (1)$$

Where $\Delta\sigma_{grain}$ is the contribution from grain boundary strengthening, $\Delta\sigma_{ss}$ is solid solution strengthening, $\Delta\sigma_{DW}$ is dislocation wall strengthening, and $\Delta\sigma_{precip}$ is precipitate strengthening. In 5xxx alloys, Mg solid solution strengthening and dislocation strengthening are the largest factors, with their weights relative to each other dependent on the specific alloy and temper. These factors will always be more important than grain boundary and precipitation strengthening in 5xxx.

Each of these features can contribute to the variability in sensitization behavior and the potential impact of sensitization on the yield strength. Fundamentally, sensitization requires the diffusion of Mg (via dislocations and grain boundaries with a rate dependence on temperature) to favorable nucleation sites (precipitates and grain boundaries) to form the β phase. Hence, the resultant size and distribution of β phase exhibits complex relationships with the initial microstructure and thermal history.

Table 2 Microstructural features of interest

Feature	Sensitization	Yield strength
Mg content	β phase fraction	Solid solution strengthening
Dislocation density	Pathway for Mg diffusion	Dislocation strengthening
Al ₆ Mn precipitates	Nucleation sites for β	Precipitate strengthening
Grain boundaries	- Nucleation sites for β - Pathway for Mg diffusion	Grain boundary strengthening

1.2.1 Magnesium Content

The Mg content of the alloy determines the equilibrium fraction of β phase; as the Mg increases, so does the amount of equilibrium β phase, see Fig. 3. This represents the maximum amount of β that *could* form in the material; however, the kinetics required for phase formation limit the volume fraction to a level much lower than equilibrium. Table 3 shows the allowable Mg (and other chemistry) ranges for the 5xxx alloys.^{1,2} From the Mg content, it would generally be expected that the susceptibility to sensitization is worse in 5059, followed by 5456, and lastly 5083. This susceptibility order is generally the case, but due to overlap in the composition ranges between alloys and the potential overall spread per individual alloy, no conclusion can be drawn on the basis of alloy composition alone.⁴ Further, even if

one material has a higher Mg content than another, the kinetic pathways for Mg to diffuse out of solid solution to a favorable nucleation site for the β phase could be lower.

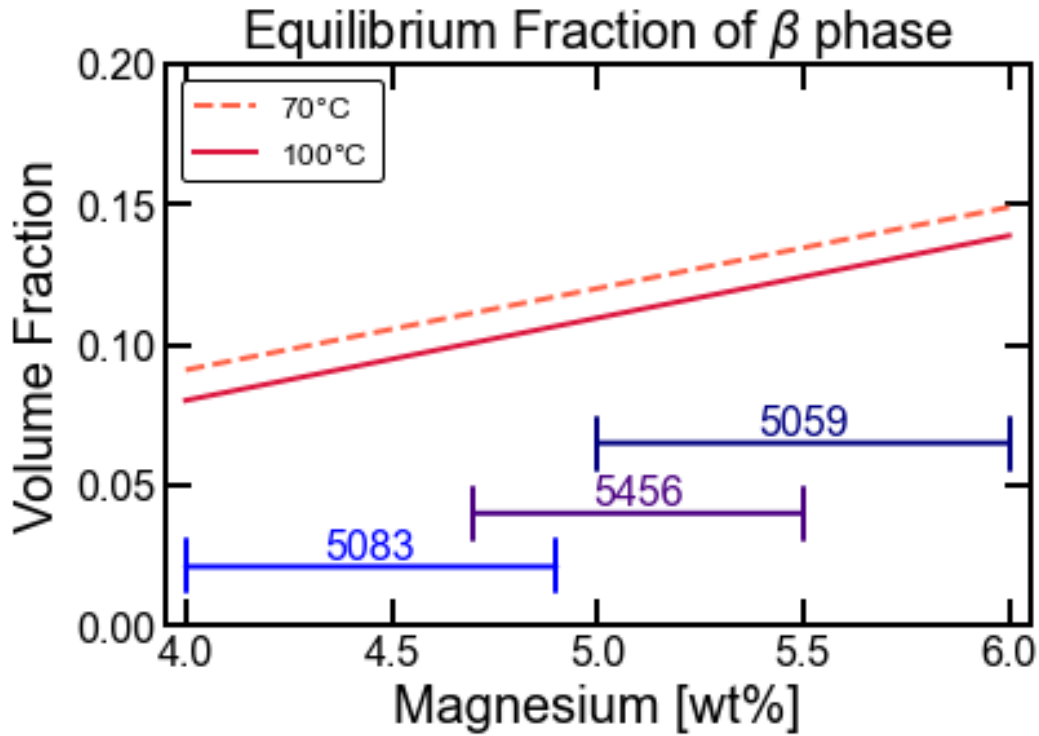


Fig. 3 Equilibrium fraction of β phase calculated using Thermo-Calc TCAL7 database¹⁵

Table 3 Composition of 5xxx alloys in wt%^{1,2}

Element	5083	5059	5456
Mg	4.0–4.9	5.0–6.0	4.7–5.5
Mn	0.4–1.0	0.6–1.2	0.5–1.0
Cr	0.05–0.25	0.25 ^a	0.25 ^a
Si	0.4 ^a	0.45 ^a	0.25 ^a
Fe	0.4 ^a	0.5 ^a	0.4 ^a
Cu	0.1 ^a	0.25 ^a	0.1 ^a
Zn	0.25 ^a	0.4–0.9	0.25 ^a
Ti	0.15 ^a	0.2 ^a	0.2 ^a
Zr	...	0.05–0.25	...

^a Maximum allowable for these elements.

From the perspective of material strength, increasing the Mg content increases the yield strength contribution from solid solution strengthening linearly⁸ as shown in Fig. 4. Generally, higher Mg content alloys will be higher strength at a given temper.

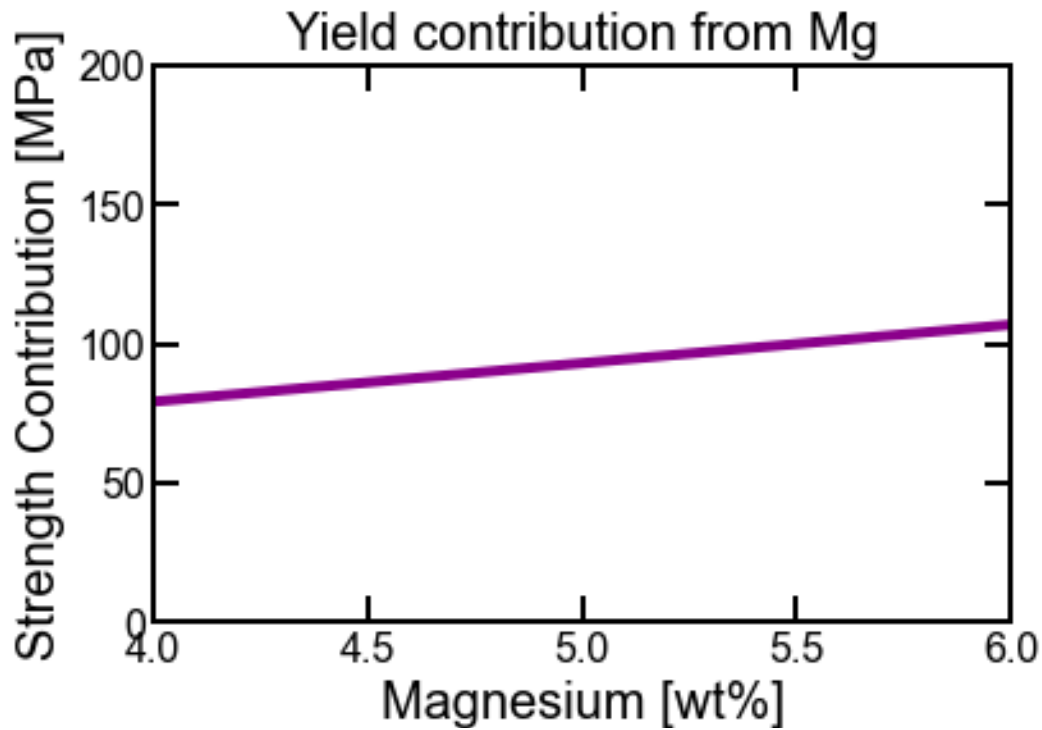


Fig. 4 Contribution to yield strength as a function of Mg content

1.2.2 Dislocations and Temper

Dislocations provide pathways for Mg diffusion (i.e., pipe diffusion) and can act as heterogeneous nucleation sites for β phase. The dislocation density in the material is increased via cold work (e.g., strain hardening imparted by rolling and/or stretching), the amount of which is described by the temper designation. Strain hardened tempers start with H and are followed by a numerical description of the degree of cold work, as described in Table 4 for two-digit tempers. Additional three-digit tempers are prevalent for specific applications. For example, the common ballistic temper is H131 and naval temper is H116. In such cases, the third digit indicates a modification to the preceding two-digit temper. Hence, a comparison of H131 and H116 indicates that the ballistic temper has higher cold work and therefore higher strength.

Table 4 Temper designation system¹⁶

Letter	First digit	Second digit
F – As fabricated		
O – Annealed		
		1 – annealed
	1 – cold worked only	2 – ¼ hard
H – Strain hardened	2 – cold worked and partially annealed	4 – ½ hard
		6 – ¾ hard
	3 – cold worked and stabilized	8 – hard
		9 – extra hard
SHTQ (Solution Heat Treated & Quenched)		

The yield strength contribution from dislocation density is calculated in Fig. 5, and helps illustrate why the H131 temper is higher strength than H116, in that the dislocation density is higher due to the higher degree of cold work. Thus, the temper designations nominally describe the degree of effective cold work and resulting strength. However, the implementation of a given temper (i.e., rolling schedules) will vary between suppliers, mills, and product thicknesses. This will result in variability within products that meet the minimum property specifications as those properties may have been attained through disparate processing histories. So, while H131 can be understood to have approximately 3× the cold work as H116, a specific temper does not translate directly to a unique microstructure or dislocation density within a given plate.

Numerous studies have indicated that susceptibility to sensitization is increased with increased cold work; for example, β phase growth rates increased with increased rolling percentages¹⁷ and the diffusion of Mg in H131 was 1.8× that of H116 given the same starting Mg content.¹⁸ Even differences within a single thick plate as a function of dislocation density/cold work have been observed, with the surface of a plate seeing more cold work and having both higher observed dislocation density and DoS.¹⁹

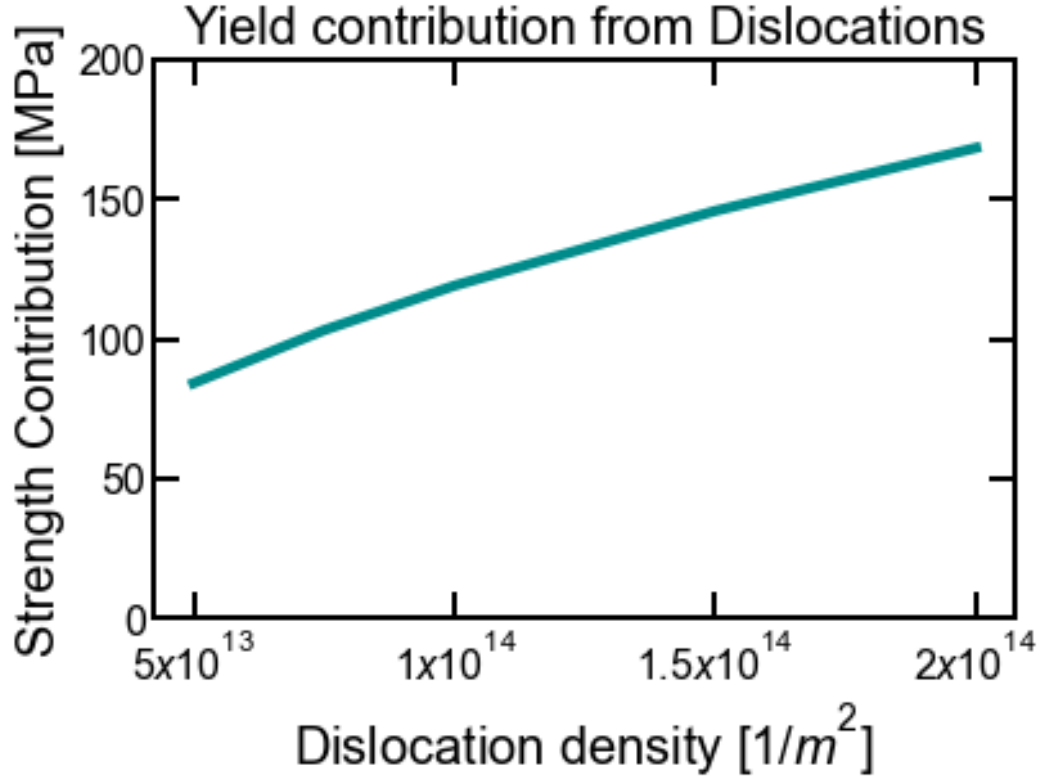


Fig. 5 Calculated strength contribution from dislocation density

1.2.3 Grain Boundaries

Grain boundaries (GBs) serve as the predominant nucleation site for the β phase. A smaller grain size results in a larger grain boundary area. Generally, this is associated with a greater number of nucleation sites and hence more β particles. Conversely, large grains with less boundary area have fewer nucleation sites, but the β particles may grow to larger sizes. In addition to the grain boundary area, the character of the boundaries can influence the nucleation and growth of the β phase; therefore, in two materials with an equivalent amount of grain boundary nucleation sites, the one with more high-angle boundaries could end up with more β phase. It is, however, still somewhat inconclusive as to the specifics of the impact of grain boundary character on sensitization despite considerable study. In Yan et al.,²⁰ high-angle boundaries were found to be more susceptible to corrosion and it was shown that the GB plane was important, not just the misorientation angle. A detailed study of precipitate growth rates found that generally a higher misorientation angle resulted in larger β phase particles;¹⁶ other studies also showed that β at high-angle boundaries had longer laths²¹ or were thicker²² than those at low-angle boundaries. In some cases, no β -phase was observed at low-angle grain boundaries²³ whereas others found a higher prevalence of nucleation at low-angle boundaries.²⁴

While it is clear that considerable variation in sensitization can occur due to grain boundary area and character, the contribution of GBs to yield strength is small. Figure 6 shows the variation in yield strength contribution due to grain size. Unless the average grain size is reduced to submicron sizes (abnormally small for commercial aluminum plates) the grain size contribution to strength will be negligible. Moreover, the contribution to strength does not vary considerably across the large range of expected grain sizes in wrought aluminum plates.

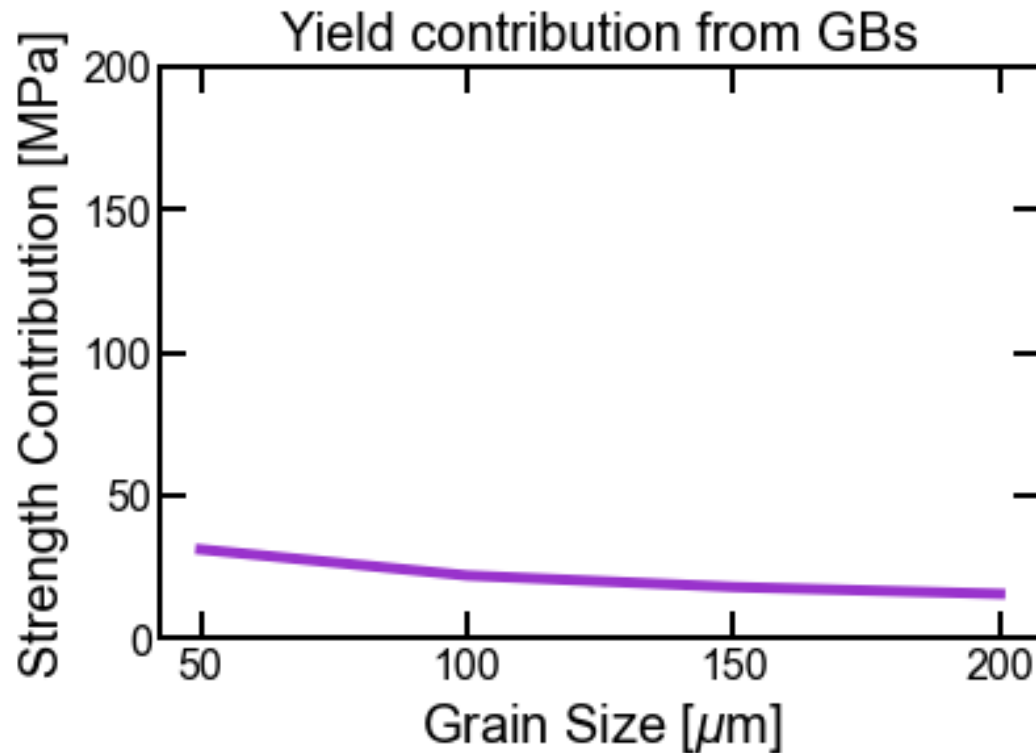


Fig. 6 Yield strength contribution from grain boundaries in 5083

1.2.4 Precipitates

Both large- and small-scale precipitate phases are present in cast and rolled 5083; the most common are Mg_2Si and Al_6Mn .¹⁶ Variability in the size and number of these phases depends on impurity content, Mn content (Table 3), and thermomechanical processing.^{16,25} While the 5xxx series of alloys is not intentionally precipitation strengthened (as in the 2xxx and 7xxx series aluminum alloys), the small-scale precipitates can have a non-negligible effect on the strength.

Huskins et al.⁸ showed about 19% of the yield strength (55 GPa) coming from the small-scale Al_6Mn precipitates. In contrast, in a different plate of 5083-H131, only 21 MPa (~7%) of the yield strength could be attributed to the precipitates, based on microstructural measurements.²⁶ Most observations of Al_6Mn size and spacing (such as Huskins et al.⁸) are performed using TEM, which has a very small field of

view. Using transmission SEM,²⁶ a much larger area can be assessed. It can be seen in Fig. 7A that the distribution of the small-scale Al_6Mn precipitates can be quite inhomogeneous, lessening the strengthening contribution. That is, the number of Al_6Mn precipitates may be high, but their spacing is large, lessening their ability to pin dislocation motion.

The strengthening contribution based on reasonable observed values is shown in Fig. 7B; while it is possible to have an appreciable impact on the strength, the precipitates are more likely to have far less of an impact than dislocation density (Fig. 5) and magnesium content (Fig. 4).

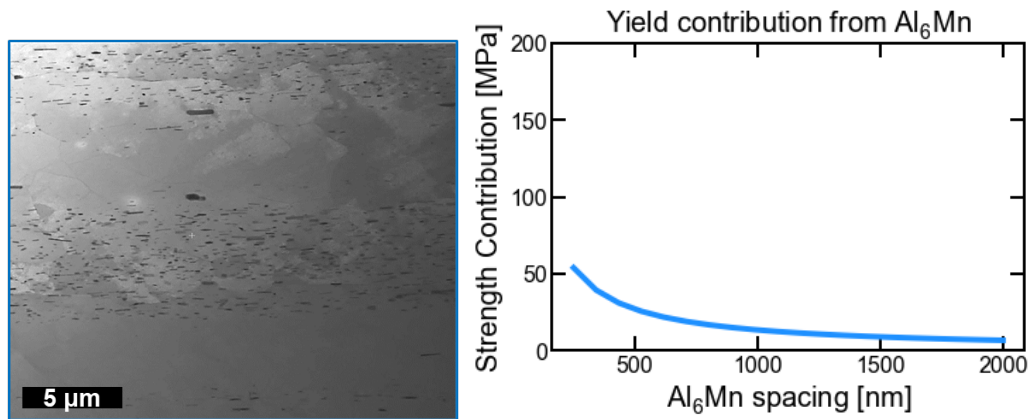


Fig. 7 (A) Transmission SEM image of small-scale Al_6Mn precipitates in an example 5083-H131 plate; and (B) likely strength contribution from small-scale precipitates

In terms of the sensitization variability, increased amounts of Al_6Mn precipitates were found to decrease β formation; however, this observation was based on high sensitization temperatures (120 and 250 $^{\circ}\text{C}$).^{25,27} More relevantly, many TEM characterization studies in sensitized materials have observed that Al_6Mn is also a favorable heterogeneous nucleation site for the β phase, in competition with the grain boundary nucleation sites. For example, β nucleated both on the Al_6Mn interface^{28,29} and on dislocations directly connected to the Al_6Mn .²² Increasing the number of favorable nucleation sites for β by increasing the Al_6Mn precipitate density would potentially increase the DoS of the material; however, as discussed in the next section, the standard test to measure DoS does not directly measure the amount of β phase and effects like this are difficult to capture.

1.3 Sensitization Test (NAMLT)

In addition to the microstructural features discussed previously, the way that sensitization is quantified also contains sources of variability. The standard test for DoS is the Nitric Acid Mass Loss Test (NAMLT) ASTM G67,^{7,30} which includes

a statement on precision as interlaboratory round robin testing indicated larger standard deviations for susceptible materials. This test immerses a standard-sized specimen in a controlled nitric acid environment; the nitric acid preferentially attacks the β phase. The sample is weighed before and after the nitric acid immersion to determine the amount of material lost.

It is important to note that, although this is a quantitative test reporting a value for mass loss, it is not a quantitative measure of the β phase itself. The location of the β skews the total mass lost through grain pullout, hence β present in sufficient quantities on a grain boundary can result in the loss of both the β and the grain while β dispersed within the volume of the grain will only be lost if it lies on the exposed surface. Figure 8 depicts this difference between inter-granular β locations versus intra-granular locations with equal amounts of β phase (in red) leading to significantly different mass loss outcomes. Critically, β within the bulk of the grains will affect the actual DoS but will not affect the NAMLT value for DoS.

This result is acceptable for the intent of NAMLT, developed to evaluate the impact of sensitization on corrosion (i.e., β on the GB causes grain pullout and more mass loss, which is worse for the intergranular corrosion and stress corrosion cracking). However, in the context of quantifying the effect of β formation on the yield strength, the DoS from NAMLT may not adequately predict the impacts on strength.

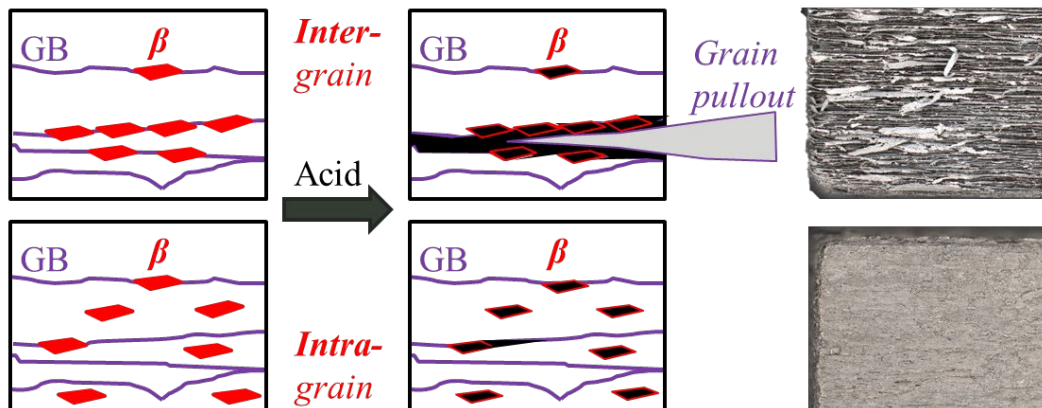


Fig. 8 Schematic of intergranular vs. intragranular β location and its effect on mass loss. Images on the right are micrographs of NAMLT samples using a Keyence VK-X1000 Series Laser Scanning Microscope.

1.4 Approach

The aim of this study is to accomplish the following:

- 1) Quantify the variability in sensitization behavior of 5083-H131 amongst suppliers.
- 2) Connect the variability in sensitization to the variability in mechanical properties of sensitized material.

Both tasks will assist in modeling the sensitization process and the impact of sensitization on performance. Increasing the availability of relevant H131 data will help with service lifetime assessments and other modeling of material behavior. In the literature review of sensitization data⁴ for commercial alloys, there were less than half the data sets for 5083-H131 than there were for H116; further, the majority of those data sets were sensitized at temperatures of 100 °C or above, which is much higher than likely service temperatures. Existing mechanical property data for sensitized 5xxx aluminum again is focused on H116 and further is almost entirely stress corrosion cracking, and other tests performed in the presence of salt water.

2. Methods

Plates of 5083-H131 were obtained from Aberdeen Test Center (ATC). Of those plates, six were initially chosen for this study with the aim of including a variety of plate thicknesses, mechanical properties, and suppliers. These plates are designated as ATC-A through -F and their thickness and Mg content (if reported) are indicated in Table 5. Data from the original 5083-H131 plate that was mechanically tested at multiple strain rates and sensitization conditions in an earlier study is also included in this report.³ To avoid confusion with an O-temper condition, the original material is designated as Omega (Ω -5083). In addition, a plate of 5083-H116 known to be highly susceptible to sensitization was provided by NSWC Carderock for comparison to the H131 temper.

Table 5 Summary of 5083-H131 plates examined in this report

Plate	Thickness (inches) ^a	Mg (wt%) ^b
ATC-A	2.75	4-4.9
ATC-B	1.8	4.58
ATC-C	2.1	4.7
ATC-D	2.5	4.7
ATC-E	1.5	4-4.9
ATC-F	2.25	4-4.9
Ω-5083	1.25	4-4.9

^a Yield strength requirement in MIL-DTL-46027M changes at a plate thickness of 2 inches.

^b Exact Mg content was not always reported, but the plates met specification; therefore, the Mg content falls between 4 and 4.9 wt% per Table 3.

2.1 Sensitization Treatments

Currently no Army specification requires a sensitization test. Consequently, two temperatures were chosen for sensitization treatments in this study, 70 and 100 °C, based on existing literature. The lower temperature has been observed in testing on a ground vehicle³¹ and the higher temperature is the standard for the Naval specification.¹ The sensitization exposure temperature of 100 °C is also the most common temperature reported in open literature.

The majority of plates were sensitized for 1 and 7 days at the two temperatures. Select plates were sensitized for longer times as well.

2.2 Tensile Test

To evaluate the effects of sensitization on mechanical properties, tensile tests were performed on each of the plates. Following MIL-DTL-46027M² and ASTM B557,³² cylindrical specimens were cut along the rolling direction with a gauge section 12.7 mm (0.5 inch) diameter and 50.8 mm (2 inches) long. The specimens included threaded ends used to attach adapters for use in hydraulic collet style grips. For plates with thicknesses of 1.5 inches and lower, specimens were cut from the centerline of the plate. For plates greater than 1.5 inches in thickness the specimens were cut from the quarterline of the plate.

An Instron servo-hydraulic universal testing machine (UTM) was used to perform the tests at a displacement rate of 0.051 mm/sec to achieve a strain rate of 0.001/s. Analog signals representing force and displacement were output from the UTM controller and collected using VicSnap with a National Instruments data acquisition system. Stereo digital image correlation (DIC)³³ was used to measure the

deformation and strain for each test. Specimens were painted with a thin coating of flat white paint followed by a flat black speckle pattern for the DIC. Typically, tests were performed within an hour of coating to minimize cracking of the surface coat during deformation. Image pairs were captured with VicSnap (Correlated Solutions) using Point Grey (Teledyne FLIR) 2.3-MP cameras with 105-mm Nikon macro lenses. Images from the tensile test were correlated using Vic3D (Correlated Solutions) to determine the strains for each test. For the determination of strain, the parameters for DIC were kept consistent for each analysis including subset and step size. Strain along the axis of the specimen was averaged over a region corresponding to a 1-inch “gauge” region. This was done to mimic the results of an extensometer as well as limit the effects of localized strain bands due to dynamic strain aging (DSA). The engineering strain data produced by DIC was used to calculate log-strain and correct for true-stress.

The strains, along with the force data, were used to generate the stress–strain curves. From these curves the yield stress was determined using 0.2% offset. Ultimate strength and failure strain were also extracted. Three specimens were tested at each sensitization condition.

2.3 DoS

The NAMLT, detailed by ASTM G67,⁷ measures the DoS of 5xxx aluminum alloys by mass loss following nitric acid (HNO₃) exposure. HNO₃ used in testing was supplied by Fisher Scientific (69%–70%). Samples, tested in triplicate, and per ASTM G67 were cut to size per specification, and ground to 320 grit with emery paper. Samples were subsequently cleaned in 5 wt% NaOH at 80 °C for 60 s, followed by HNO₃ for 30 s to desmut, rinsed in deionized water, and dried for initial mass. Subsequently, samples were placed in HNO₃ (at 30L/m³), and fully immersed in a water bath at 30 °C for 24 h. Following the 24 h, samples were double rinsed in deionized H₂O and, using a stiff plastic bristle brush, brushed under deionized H₂O to remove any loose grains. Samples were then subsequently dried with lab nitrogen, allowed to air dry overnight, and massed to calculate and report mass loss as DoS.

The mass loss can be related to the kinetics of β phase formation via the following modified Johnson–Mehl–Avrami–Kolmogorov (JMAK) model:⁴

$$m(t) = (m_{max} - m_0)(1 - e^{-kt^n}) + m_0 \quad (2)$$

Where m is the mass loss at time t , m_0 is the initial mass loss and m_{max} is the saturated mass loss (e.g., all the Mg that would transform to β has done so). The experimental data was fit to Eq. 2 using Python and applying bounds for some of

the parameters; the initial mass loss, m_0 , was limited to an upper limit of 15 mg/cm², which is the maximum DoS value for “resistant” to sensitization in ASTM G67. The exponent, n , is bounded between 0.5 and 2.5, which correspond to the allowable types of nucleation and morphology for precipitation.⁴ The saturation, m_{max} , is bounded between 15 to 100 mg/cm².

2.4 Microstructural Characterization

2.4.1 Quantification of β Phase on Grain Boundaries

The characterization approach to quantify the amount of β phase on the grain boundaries is based on the work of McMahon et al.³⁴ and involves performing both scanning electron microscopy (SEM) and electron backscattered diffraction (EBSD) on the same region of sample. Three areas of size approximately 200 × 200 μ m were examined on each of the three sensitization conditions of the Ω -5083-H131 plate: as-received, 7 days at 100 °C, and 28 days at 100 °C. The areas were tracked with the use of fiducial markers placed with the focused ion beam.

First, an EBSD scan is performed of the area to identify grain boundaries. The sample area is prepared for the EBSD scan via standard metallographic polishing techniques. The scans were performed on a FEI NanoLab 600 using an EDAX EBSD camera. Scan cleanup was performed with OIM software using grain dilation cleanup with a grain tolerance angle of 5, a minimum grain size of 5 points, and a minimum confidence index of 0.1.

Next, the surface is etched with 10% ammonium persulfate solution for approximately 30 min; details of the etching process can be found in McMahon et al.³⁴ The etch attacks the β phase. The area is imaged with the SEM and the SEM and EBSD images are overlaid in the image analysis software ImageJ. The SEM image with the etched areas corresponding to the location of β phase is segmented using thresholding and binarized to highlight those areas. In comparison with the locations of the grain boundaries from the EBSD map, the percentage of grain boundary that had β phase present can be calculated. This process is illustrated in Fig. 9.

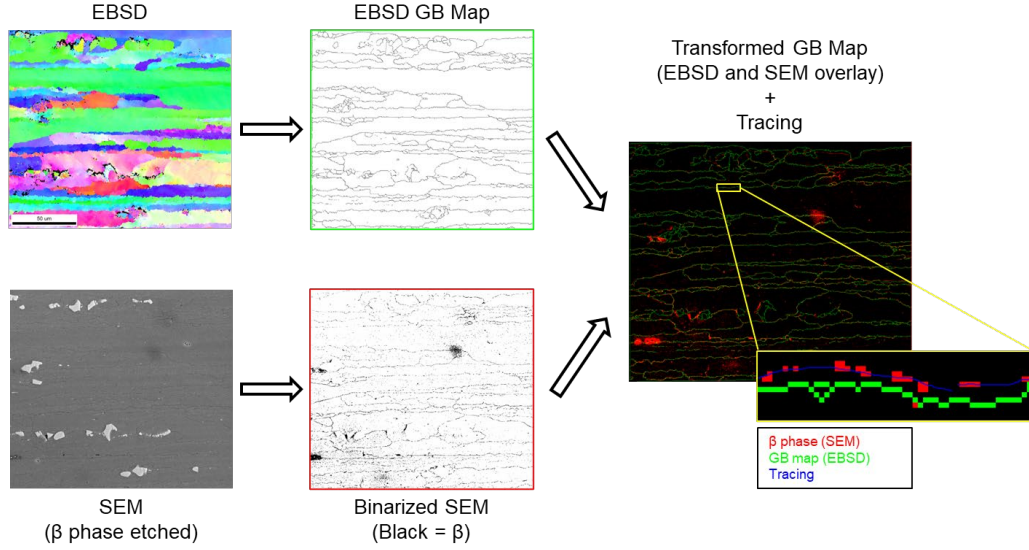


Fig. 9 Schematic depicting the multistep analysis of quantifying β phase located at the grain boundaries

2.4.2 Nanoscale Characterization

Electron transparent foils were prepared for analysis via transmission SEM and transmission electron microscopy (TEM) via twinjet electropolishing. The 3-mm disks were ground to a thickness of about 100 μm . Twinjet electropolishing was performed in a 25% nitric acid in methanol solution cooled to less than -30°C at 7 to 10 V.

After twin jet polishing, the foils were observed via transmission SEM or TEM. The SEM observations were made in a ThermoFisher Apreo S at 30 kV using a retractable STEM detector. TEM was performed using a JEOL 2100 at 200 kV operating in STEM mode.

3. Results and Discussion

First, the DoS was quantified for the plates at varying sensitization times and temperatures via NAMLT. The tension tests on as-received and sensitized samples are then correlated with the DoS and further with the microstructural features relevant to the mechanisms of yield strength in the material.

3.1 NAMLT

The DoS measured by the NAMLT test for the seven plates in this study are recorded in Table 6 and plotted in Fig. 10. The lines in Fig. 10 are fits to the DoS values using the JMAK kinetic model of precipitation nucleation and growth, Eq. 2. There is no significant variation in the as-received DoS values (given the

standard error bars for NAMLT of ± 4 per the interlaboratory test of unsensitized material found in ASTM STP 516).^{7,30}

Table 6 Degree of sensitization from ASTM G67

Condition		Mass loss (mg/cm²)							Ω	H116
		ATC-A	ATC-B	ATC-C	ATC-D	ATC-E	ATC-F			
As-received		3	6	2	3	4	4	5		2
100 °C	1 day	10	9	7	7	8	11	8		9
	3 days	...	17	...	...	13	...	...		...
	7 days	40	23	25	32	22	29	23		25
	14 days	37	28	...	...	40	...	...		69
	28 days	39	38	...	...	51	...	32		...
70 °C	1 day	4	6	3	3	5	6	...		3
	3 days	...	7	...	...	5	...	...		...
	7 days	7	13	5	5	6	8	...		4
	14 days	...	13	...	...	8	...	...		7
	28 days	...	17	11	...	11	...	...		25
	105 days	...	...	...	...	...	...	...		34

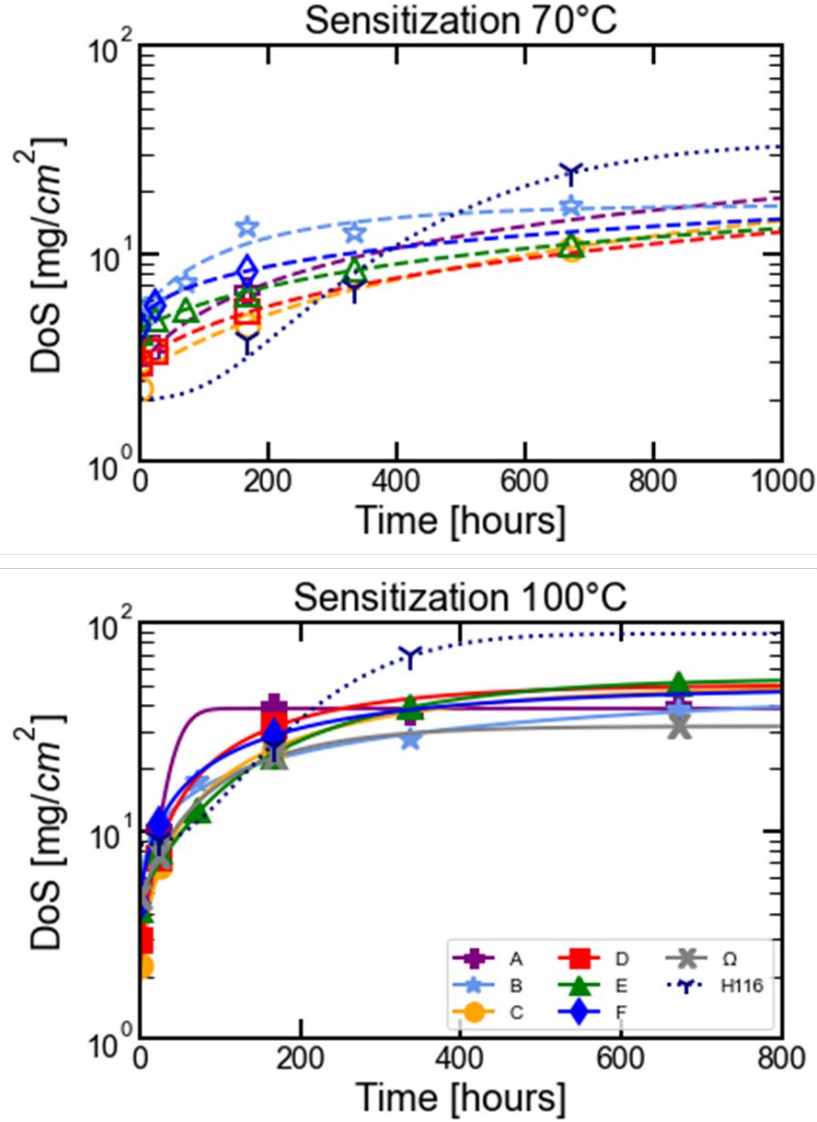


Fig. 10 DoS at 70 and 100 °C as a function of exposure time

As expected, sensitization levels at 70 °C exposure are much lower than those at 100 °C for all plates. Variability in sensitization behavior is more apparent at longer exposure times and with the kinetic model fits included. For example, ATC-A reaches a high DoS rapidly, exhibiting the largest value of the samples exposed to 100 °C for 1 week; however, it also saturates at that value, such that after long exposure times it actually has a lower DoS than the other plates. The 70 °C sensitization levels tend to saturate lower than the saturation levels at 100 °C. Some of the plates sensitized at 70 °C would not be expected to reach the threshold for sensitization (25 mg/cm²) set forth by ASTM G67 even at extremely long times, whereas all of the plates sensitized at 100 °C reach that threshold within 2 weeks or less.

Interestingly, the H116 plate reaches higher DoS values than any of the H131 plates at long times despite the general expectation of lower susceptibility owing to the lower degree of cold work. In fact, the kinetics are slower, as is more obvious in the sensitization at 70 °C, which is the aspect more directly related to the cold work. However, there is a high fraction of GBs in this plate,³⁵ increasing the sites for nucleation and overall proportion of β phase.

3.2 Tensile Behavior

The yield and ultimate tensile strength at two sensitization conditions for each of the plates are shown in Table 7. The initial properties of the plates in the as-received condition show some variability. An example tensile curve from the as-received ATC-A is shown in Fig. 11, with the inset highlighting the serrated flow from DSA.^{36–38} DSA is a consequence of Mg solute interacting with the mobile dislocations under certain loading conditions (strain rates and temperatures) resulting in serrated flow. The localized deformation bands associated with the serrations are also clearly visible in the DIC analysis. Fracture occurs on planes directly associated with the DSA deformation band, mostly on a single plane 45° to the axis of loading but occasionally on a pair of perpendicular 45° planes. There was no observed difference in failure mode between the as-received or sensitized samples.

Table 7 Mechanical properties of 5083-H131

Condition	Yield strength (MPa)						Ω
	ATC-A	ATC-B	ATC-C	ATC-D	ATC-E	ATC-F	
As-received	293.9 ± 0.27	280.17 ± 0.94	289.93 ± 1.18	296.3 ± 0.41	286.96 ± 0.18	295.49 ± 0.2	283.06 ± 0.38
1 day at 100 °C	246.9 ± 0.76	239.02 ± 0.53	249.52 ± 2.52	256.57 ± 0.63	250.84 ± 0.78	250.02 ± 0.16	245.12 ± 1.15
7 days at 100 °C	...	...	...	...	...	...	222.43 ± 0.95
7 days at 70 °C	262.16 ± 1.06	254.53 ± 0.31	263.05 ± 1.01	270.07 ± 1.13	265.32 ± 0.87	264.94 ± 0.32	...
Condition	UTS (MPa)						Ω
	ATC-A	ATC-B	ATC-C	ATC-D	ATC-E	ATC-F	
As-received	348.35 ± 1.74	344.57 ± 0.36	354.70 ± 0.73	365.03 ± 0.44	360.30 ± 0.70	350.52 ± 0.27	390.19 ± 1.54
1 day at 100 °C	337.42 ± 0.89	339.76 ± 0.35	348.66 ± 1.41	359.13 ± 0.38	355.79 ± 0.21	341.31 ± 0.53	383.87 ± 2.47
7 days at 100 °C	...	...	...	...	...	...	390.50 ± 0.82
7 days at 70 °C	332.89 ± 0.82	335.24 ± 0.29	346.72 ± 2.11	355.91 ± 0.50	351.60 ± 0.60	337.66 ± 0.60	...

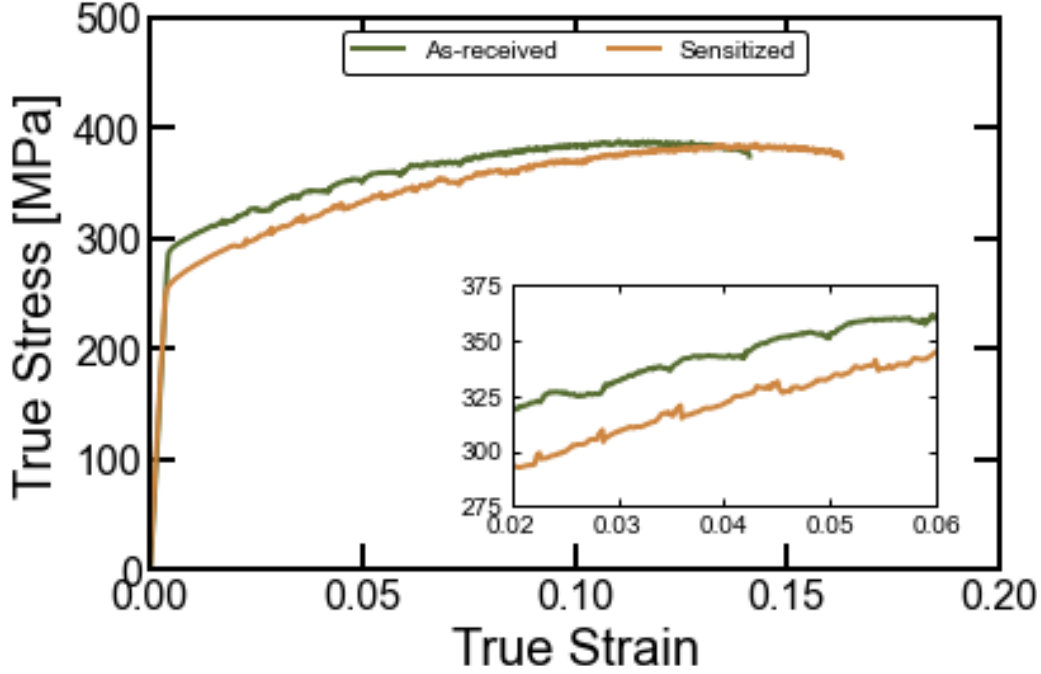


Fig. 11 Example stress–strain curves for ATC-A 5083-H131 in the as-received and 7 day at 70 °C sensitized condition with inset highlighting the serrated flow from DSA

All plates exhibit some drop in yield as a result of sensitization, with higher drops associated with the higher temperature conditions. The ultimate tensile strength (UTS) is less affected by sensitization; as can be seen in Fig. 11, although the yield of the sensitized curve is lower, with strain hardening, the UTS ends up near the same level of stress.

The Johnson–Cook (JC)³⁹ plasticity model is commonly used in the study and simulation of deformation of metals. The simple functional form of the JC model is amenable for incorporating into finite element models to describe materials. It is an empirical model consisting of three parts. The first is a strain hardening term that defines the flow stress as a function of plastic strain (ϵ_{pl}). The second and third parts consist of a strain rate ($\dot{\epsilon}$) and temperature (T) dependent relations, respectively. The flow stress is in the form of Ludwik strain hardening consisting of three terms, A , B , and n .

$$\sigma_y = (A + B\epsilon_{pl}^n)(1 + C \ln \dot{\epsilon}^*)(1 - T^{*m}) \quad (3)$$

By analyzing data at the reference strain rate and room temperature, the first part may be isolated, and the A , B , and n terms can be determined for each plate and condition. The tensile curves for the plates in the as-received and sensitized conditions are fit to this first part, with the resulting parameters in Table 8. The parameters are also plotted in Fig. 12A to highlight the differences. In Fig. 12B, the

average values of A , B , and n for each sensitization condition are used to generate the stress–strain curve with a confidence interval based on the spread of the parameters. From these plots it is clear that there are significant differences between the sensitized and unsensitized conditions.

Table 8 Johnson–Cook strength parameters

Condition Plate	As-received			7 days at 70 °C			1 day at 100 °C		
	A (MPa)	B (MPa)	n	A (MPa)	B (MPa)	n	A (MPa)	B (MPa)	n
ATC-A	247.67	260.09	0.2945	210.88	320.67	0.3111	192.86	350.32	0.3156
ATC-B	250.33	336.08	0.4132	216.69	385.97	0.3953	194.69	407.23	0.3764
ATC-C	252.97	322.60	0.3793	218.58	379.54	0.3752	203.18	403.00	0.3719
ATC-D	261.66	351.24	0.3958	223.74	393.23	0.3664	209.24	422.96	0.3734
ATC-E	246.02	360.76	0.3695	218.74	405.94	0.3656	196.74	427.72	0.3508
ATC-F	259.18	278.28	0.3448	223.91	347.68	0.3641	200.83	366.10	0.3390
Ω	246.98	315.54	0.3697	...	...	...	198.43	434.21	0.3649
Average	252.12 ± 5.71	317.80 ± 34.26	0.367 ± 0.036	218.76 ± 4.43	372.17 ± 29.10	0.363 ± 0.026	199.42 ± 5.16	401.65 ± 29.58	0.356 ± 0.021

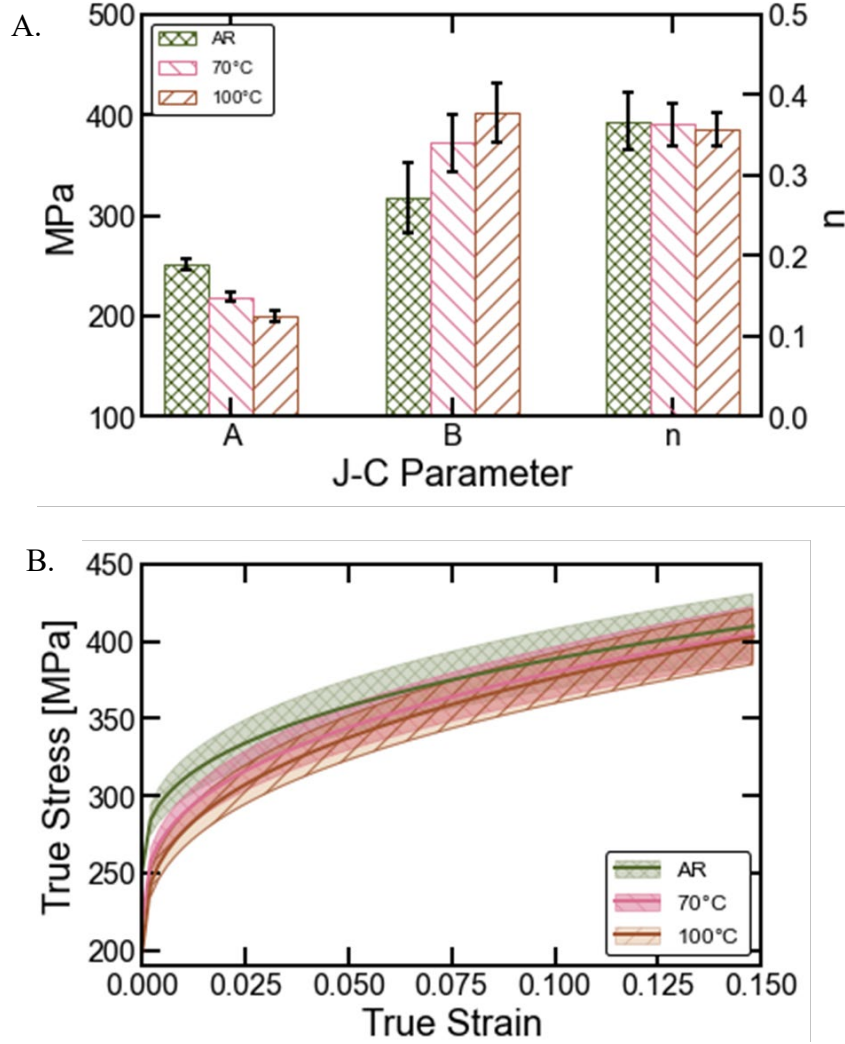


Fig. 12 (A) Average Johnson–Cook parameters A , B , and n for the as-received and two sensitized conditions; and (B) example stress–strain curves generated from these parameters

The decrease in yield strength with sensitization is clearly reflected in the decrease in the A parameter. The limited impact of sensitization on the UTS can be understood in the context of the corresponding increase to B , which is proportional to the strain hardening rate ($d\sigma/d\epsilon$). Considere’s criterion states that the onset of necking (and consequently the UTS) occurs when $\sigma = d\sigma/d\epsilon$. Using the fit JC parameters in Table 8 to calculate the uniform strain using this criterion indicates that there would be an increase from 0.14 to 0.19 for the AR and 100 °C sensitized condition and that the corresponding UTS would increase slightly. Hence, while the yield strength (A) decreases with sensitization, this decrease is offset by the larger increase in B . While n also impacts the UTS, the differences in this parameter between the plate conditions are negligible. In practice, the uniform strain is found to increase with sensitization (Fig. 11), yet the UTS decreases slightly. This likely stems from the apparent saturation in hardening observed in all samples that is not

well captured by the JC model. Furthermore, as the UTS is not strongly affected by sensitization the focus of the remainder of the report will be on yield strength, which shows significant differences.

In order to quantify the impact of sensitization on the yield strength, an empirical fit to the change in yield strength is made. This is a first approximation without mechanistic explanation. We then perform a more rigorous analysis of the yield drop based on the available deformation mechanisms.

The sensitized tensile data in Fig. 13 is fit with an equation of the form:

$$\Delta\sigma_y = A * DoS + \frac{B}{DoS} + C \quad (4)$$

Where $\Delta\sigma_y$ is the change in yield strength (absolute change or percent change) and A, B, and C are the fitting parameters. The fit parameters for both the absolute and percentage change in strength are summarized in Table 9; the ATC-B plate sensitized at 70 °C was not used for the fit.

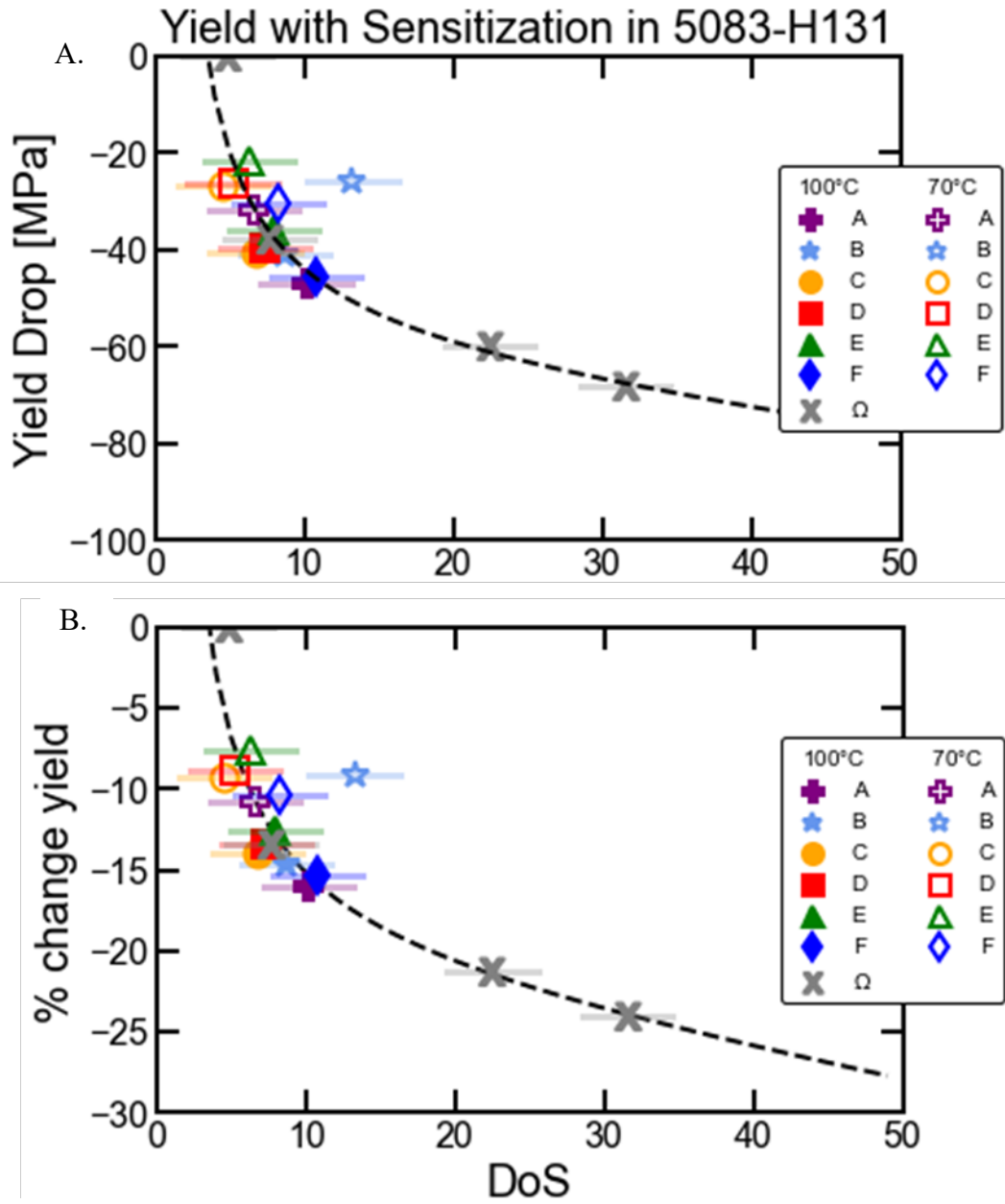


Fig. 13 (A) Absolute yield drop in MPa as a function of DoS (B); percent drop in yield as a function of DoS; lines are the empirical fits

Table 9 Empirical fit parameters

Yield drop type	A	B	C
<i>Absolute H131</i>	-0.39569442	221.17899201	-62.10102393
<i>Percent H131</i>	-0.16839973	75.13986971	-20.99720704

The mechanical properties of sensitized 5083-H116 temper are compared to that of H131; since this is only one plate, we do not quantify the empirical fit. However, it is clear that the yield drop in sensitized H116 is less than that of the H131 plates at the same DoS. While the initial yield strength of the H116 plate is lower, as expected, the lower impact of sensitization on yield strength trend is clear whether considering either absolute yield drop or percent change in yield (Fig. 14). The measured mechanical properties values are shown in Table 10. Similar to the sensitized H131 plates, the UTS in the H116 temper does not change significantly for the sensitized conditions.

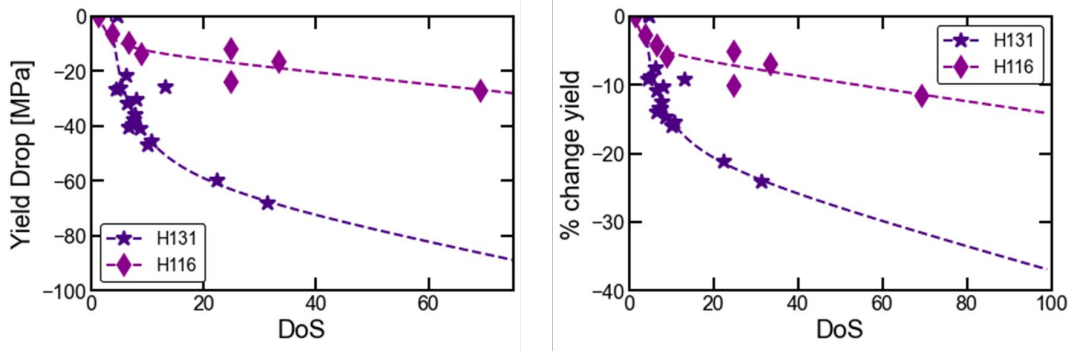


Fig. 14 Comparison of absolute yield drop in MPa between H131 and H116 tempers

Table 10 Mechanical properties of sensitized 5083-H116

Condition		Yield [MPa]	UTS [MPa]
As-received		234.83 ± 3.27	329.97 ± 1.25
70 °C	7 days	228.12 ± 1.63	327.97 ± 1.03
	14 days	224.89 ± 2.22	327.28 ± 1.01
	28 days	222.89 ± 0.90	328.11 ± 1.01
	105 days	218.15 ± 1.46	329.23 ± 0.65
100 °C	1 day	220.93 ± 3.26	327.00 ± 1.37
	7 days	210.90 ± 1.43	326.62 ± 1.39
	14 days	207.76 ± 1.03	324.89 ± 0.76

3.3 Mechanisms of Yield Drop

The dual impacts of key microstructural features on both sensitization and yield strength were listed briefly in Table 2 of Section 1.2 and will be described in detail herein. The yield strength can be estimated by a linear combination of contributions from different strengthening mechanisms, as was shown in Eq. 1, repeated here for ease of reference:

$$\sigma_{yield} = \Delta\sigma_{grain} + \Delta\sigma_{SS} + \Delta\sigma_{DW} + \Delta\sigma_{precip} \quad (1)$$

Where $\Delta\sigma_{grain}$ is the contribution from grain boundary strengthening, $\Delta\sigma_{ss}$ is solid solution strengthening, $\Delta\sigma_{DW}$ is dislocation wall strengthening, and $\Delta\sigma_{precip}$ is precipitate strengthening. The contributions can be represented by relationships with the quantitative measures of the microstructural features themselves:

$$\sigma_{yield} = kd^{-1/2} + HC^\alpha + \tilde{k}l^{-1/2} + \sqrt{3}\frac{Gb}{L} \quad (5)$$

Where d is the grain size, C is the composition of Mg in weight percent, l is the dislocation wall spacing, and L is the spacing between precipitates. The remaining terms are material specific constants that will be described in the following sections. In the following sections we will attempt to specifically quantify those mechanisms based on the experimental observations mostly from the Ω -5083-H131 plate.

3.3.1 Magnesium Solid Solution Strengthening

As the Mg leaves solid solution to form β phase during the sensitization process, it will reduce the yield strength according to

$$HC^\alpha \quad (6)$$

Where C is the composition of Mg in weight percent. H and α are material constants with the values used for Al-Mg from Huskins et al.⁸

While the relationship between the Mg in solid solution and the strengthening contribution it provides is straightforward, quantifying the composition of Mg in its possible locations (e.g., in solid solution vs. in the β phase) is challenging. Light elements such as Mg are notoriously difficult to quantify using characterization methods such as energy-dispersive X-ray spectroscopy (EDS). Further, the Mg will be inhomogenously distributed, traveling along dislocations and forming depleted regions around β particles.⁴⁰

The absolute upper bound of Mg leaving solid solution would be the equilibrium composition of β phase at the given sensitization temperature. The concentration of Mg left in solid solution after the equilibrium amount of β phase has precipitated would be 1.28 wt% at 100 °C and 0.88 wt% for 70 °C, according to the phase diagram calculated using Thermo-Calc TCAL7.¹⁵ The drop in yield strength associated with this change in Mg content is in Fig. 15 for the range of Mg content specified for AA5083. The maximum yield drop is larger for 70 °C as the equilibrium fraction of β is higher at lower temperatures (the solubility of Mg in solid solution decreases as temperature decreases). However, the kinetics will be far slower at 70 °C than 100 °C. As a result, alloys sensitized at 70 °C are less likely to attain the equilibrium β fraction.

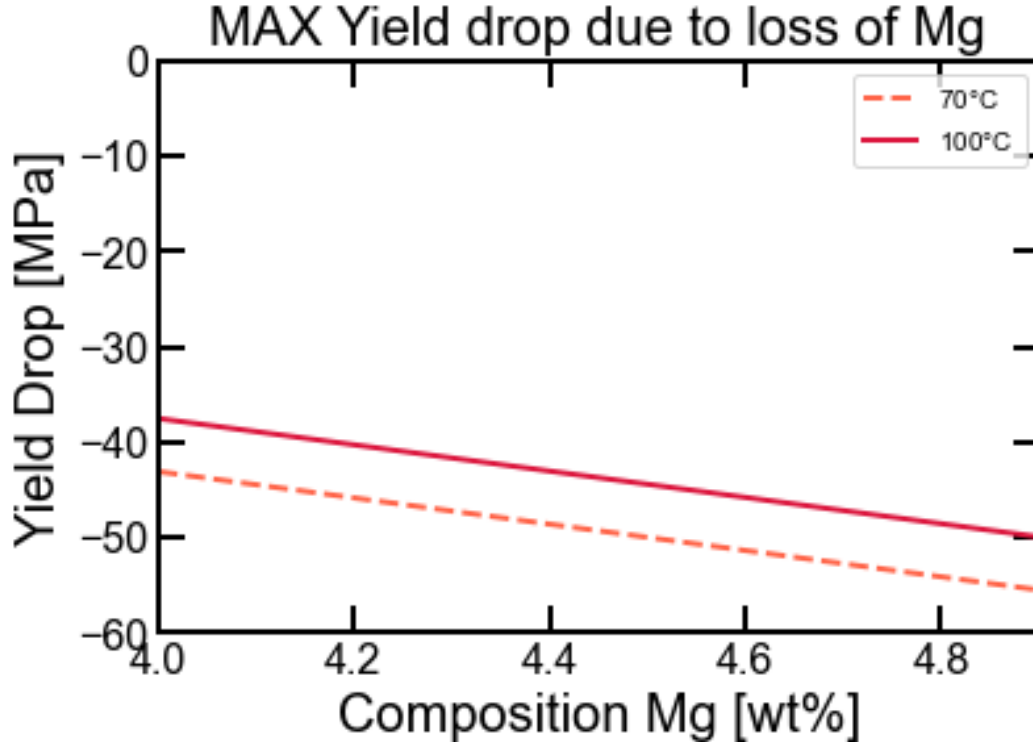


Fig. 15 Maximum yield drop possible if the equilibrium fraction of β phase is formed

Given the sluggish kinetics of Mg diffusion at low temperatures it is unphysical for the material to reach equilibrium β phase fraction. Sensitization studies out to several months have never observed anywhere near the equilibrium fraction; after 30 months at 70 °C the volume fraction of β was only measured at 0.6%⁴¹ where the equilibrium volume fraction would be 11%. However, we establish the equilibrium state as an absolute upper bound for yield drop due to loss of Mg from solid solution.

The existing experimental measurements of Mg concentration in solid solution, while limited, show a much smaller decrease, particularly at lower temperatures. Goswami and Holtz⁴² used fine-probe EDS in TEM observations of 5083-H116 sensitized at 100 °C and their results are reproduced in Fig. 16. It can be seen that there is a wide variation in measurements even within the as-received material, but there is a general downward trend in the Mg concentration with increasing time. Sampath et al.⁴³ estimated the Mg solid solution content via electrical conductivity in 5083-O. They reported the time to reach a Mg content of 4 wt% (down from initial value of 4.5 wt%) at several temperatures. Yi et al.⁴¹ measured the matrix Mg content in 5083-H116 at 50 °C for 24 months using atom probe tomography (APT) to be 4.51 wt% down from an initial 4.75 wt%. They also performed precipitation modeling for both 5083-H116 and H131 at 70 °C, which included the evolution of matrix Mg composition as part of the modeling results; their

predictions generally agreed well with the observed precipitate size and distribution, so it is assumed that their predictions for the Mg content are also generally reasonable. Their prediction for 1 week at 70 °C is 4.72 wt% (down from 4.75 wt%).

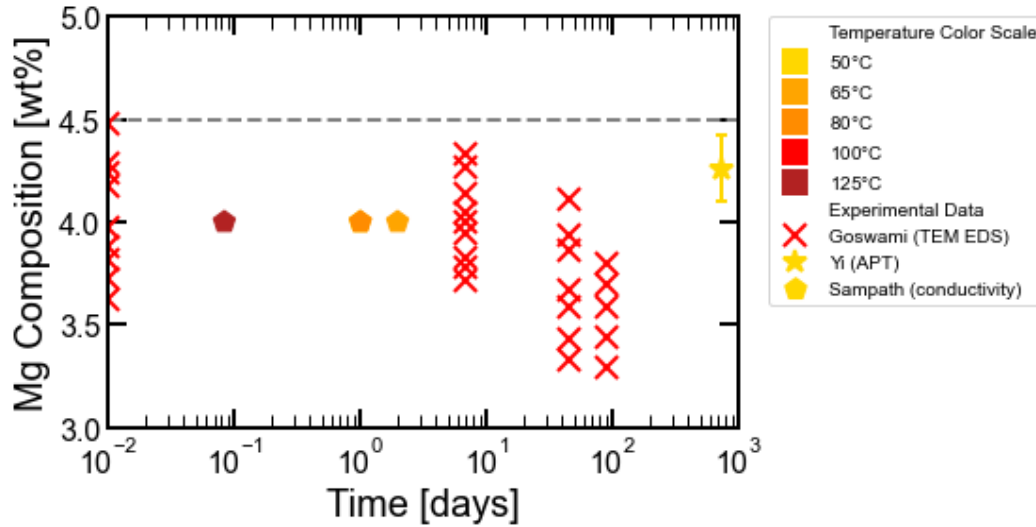


Fig. 16 Literature data for decrease in Mg content in solid solution with sensitization. Exposure temperature for sensitization is indicated by color scale. Decrease is scaled to an initial Mg value of 4.5 wt%.

Given the lack of data in this area and the challenge of quantifying Mg directly, we attempt a reasonable estimate based on detailed characterization of the microstructural features using the Ω -5083-H131 plate. The prevailing feature of interest would seem to be the formation of β phase on the grain boundaries. This is the most studied aspect of sensitization as β precipitation at the GB contributes to IGC, subsequent IGSCC, and in-service cracking issues (for the Navy). However, the β phase can also nucleate in the grain interior, generally on dislocations or boundaries of existing precipitate phases, such as Al_6Mn (heterogenous nucleation). This has been underreported/understudied in literature. Figure 17A shows a TEM image with β phase precipitates on the GB while Fig. 17B shows their nucleation on the Al_6Mn interface. A third possibility is the segregation of Mg to dislocations, as evidenced in Fig. 17C by the bright decoration on the dislocations particularly visible throughout the grain in the upper half of the image. The segregation of Mg to dislocations has been studied mostly in the context of DSA, in order to understand the mechanisms of interaction during deformation.

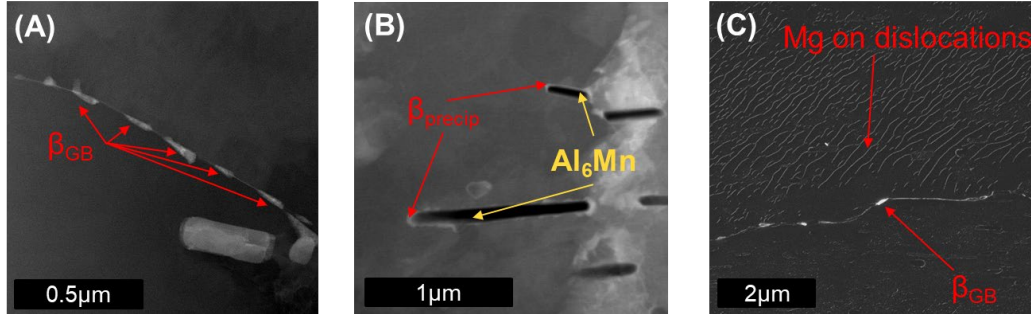


Fig. 17 Representative images of possible locations of Mg once it has left solid solution: (A) β phase formed on grain boundaries; (B) β phase formed on Al_6Mn precipitates; and (C) Mg decorating dislocations

We will make efforts to generate reasonable estimates of these three mechanisms based on advanced microstructural characterization. The quantification of β on grain boundaries can be achieved through combined SEM and EBSD scans, which enables a more representative volume of material as compared to the TEM foils necessary for examination of Al_6Mn and dislocation features.

3.3.1.1 Mg/ β on Grain Boundaries

We first quantify the amount of β on the GB as a lower bound of the amount of β . The methodology was described in Section 2.4.1, adapted from McMahon et al.³⁴ In McMahon et al.³⁴ it was found that increasing DoS was correlated with increasing GB coverage; however, between samples of different alloy or temper, the same DoS value from NAMLTL actually had very different amounts of β phase on the grain boundaries. For example, comparing samples of 5086-H116 and 5456-H116 that both had a DoS of 24 mg/cm², the measured GB coverage of β was 64% and 27%, respectively.

The results for the Ω -5083-H131 plate sensitized at 100 °C for 7 and 28 days are in Table 11. Using these values, the volume percent of β present at the GB in the material at each condition can be estimated, and the amount of Mg that is present in the β at the GB rather than in solid solution can be calculated. These values are in Table 11, along with the associated drop in yield. We will take this as the *minimum* loss of Mg from solid solution that has formed β since this is a (mostly) direct observation of the phase itself. It is the minimum Mg lost because we know there is also Mg in the β phase nucleated intragranularly (e.g., on Al_6Mn) and on the dislocations.

Table 11 Measurements of GB and β at GB in Ω -5083-H131

Sensitization condition	GB % (area percent)	β % of GB (area percent)	Mg in GB β (wt%)	drop in yield (MPa)
AR	2.9	9.5 ± 0.3	0.1033	...
7 days at 100 °C	2.2	22.4 ± 2.7	0.0815	1.125
28 days at 100 °C	2.4	55.4 ± 1.1	0.3953	5.455

3.3.1.2 β on Al_6Mn Particle Interfaces

A representative micrograph of β phase nucleating on a small Al_6Mn precipitate is shown in Fig. 18. (This is a HAADF image, so it has the alternate contrast of Fig. 17; i.e., the lighter element, Mg, will be darker.) From large-scale images (see Fig. 7), the area fraction of the small-scale Al_6Mn particles in the Ω -5083-H131 plate is 3.62% and the average area is $0.259 \mu\text{m}^2$ (from average ellipsoid measurements of $0.55 \mu\text{m}$ major axis and $0.15 \mu\text{m}$ minor axis).

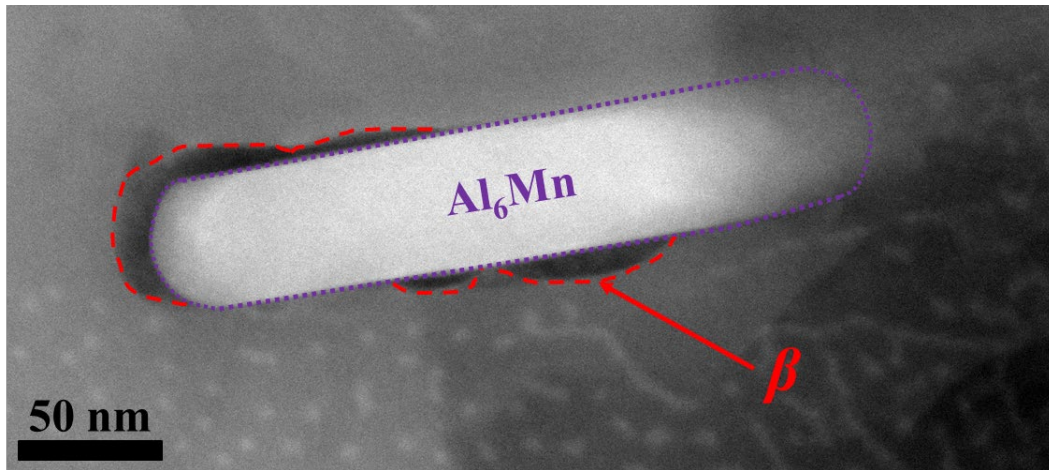


Fig. 18 TEM micrograph of β phase nucleating on a small-scale Al_6Mn precipitate

By assuming that some fraction of the Al_6Mn precipitates have β nucleated on them, and that the size of this β is a fraction of the size of the Al_6Mn , we can then estimate the amount of Mg in the β phase that is present on the nanoscale precipitates. An approximated upper bound would be that all of the Al_6Mn precipitates have β nucleating on them and that the size of the β phase is half that of the Al_6Mn . Based on TEM and SEM observations, a more reasonable estimate would be that between 10% and 50% of Al_6Mn support some nucleated β phase and that the size of the β phase is between 10% and 50% of the Al_6Mn size. The resulting drops in yield stress for all estimates of β phase on Al_6Mn are included in Table 12.

Table 12 Estimations of β on Al₆Mn in Ω -5083-H131

Estimated % of Al ₆ Mn with β	Estimated β size % of Al ₆ Mn	Mg in β on Al ₆ Mn (wt%)	Drop in yield (MPa)
100	50	0.6788	9.367
50	50	0.3375	4.658
10	10	0.0136	0.188

3.3.1.3 Mg on Dislocations

Mg is known to segregate to dislocation cores due to a combination of faster pipe diffusion (compared to bulk diffusion) and strong dislocation-solute interactions.³⁶ Figure 17C showed the Mg located on dislocations by contrast (lighter/brighter = Mg) in the Ω -5083-H131 sample sensitized at 100 °C for 7 days.

Both experimental observations (APT)³⁶ and modeling predictions⁴⁴ have investigated the interaction of Mg with dislocations. There is clear enrichment of Mg in one location on the dislocation, that is, the bright concentrations observed in Fig. 17C, but there are also depleted regions of Mg in other locations of the stress field surrounding the dislocation. In aggregate then, across the region of influence around a dislocation, the Mg is not lost from solid solution, merely redistributed around the dislocation core. Therefore, the indications of Mg segregation observed in the sensitized material (Fig. 17C) are not necessarily indicative of overall loss of solute from the solid solution.

While the solid solution strengthening is quantified via Eq. 6, this term encapsulates the impact of the substitutional atoms (Mg) on the movement of dislocations through the matrix (Al). While the overall amount of Mg present may not be affected by the clustering at dislocations, the mechanism of interaction with dislocations may be.

In Al-Mg alloys, the strong interactions of Mg with dislocations and fast diffusion give rise to dynamic strain aging (DSA) and the related Portevin–Le Chatelier effect (PLC). DSA occurs when the Mg solute atoms are sufficiently mobile to repeatedly pin mobile dislocations as the dislocations glide through the matrix, leading to serrated flows. The occurrence of DSA depends strongly on the imposed strain rate, solute concentration, and initial dislocation density.^{45–47}

Due to the dependence of DSA on solute concentration and initial dislocation density, changes in interaction of Mg with dislocations between the as-received and sensitized samples can be observed via the serrated flow in the stress–strain curves, Fig. 19. Here, the changes in PLC effect appear mainly in the evolution of critical strain for the onset of serrations, but also their spacing and stress drop. As can be

seen in Fig. 19, the onset of serrated flow in the sensitized samples is significantly later than in the as-received sample. The critical strain exhibits a complex dependence on solute concentration, dislocation density, and to a lesser extent strain rate. A relation between the solute concentration and the critical strain is

$$\varepsilon_C \propto \frac{1}{L} \left(\frac{C_1}{\alpha C_0} \right)^{3/2} \quad (7)$$

Where C_0 is the solute concentration in the matrix, C_1 is the solute concentration necessary to pin the dislocation, and L the obstacle (dislocation) spacing; α is a materials constant.⁴⁸ This suggests that the critical strain should increase as Mg leaves solid solution (C_0 decreases) to form β phase. Experimental studies on Al-Mg alloys with varying global solute concentration have observed that the critical strain is likely to increase slightly with decreasing Mg content.^{45,46,49} However, this is a small effect at the strain rate used in this study.

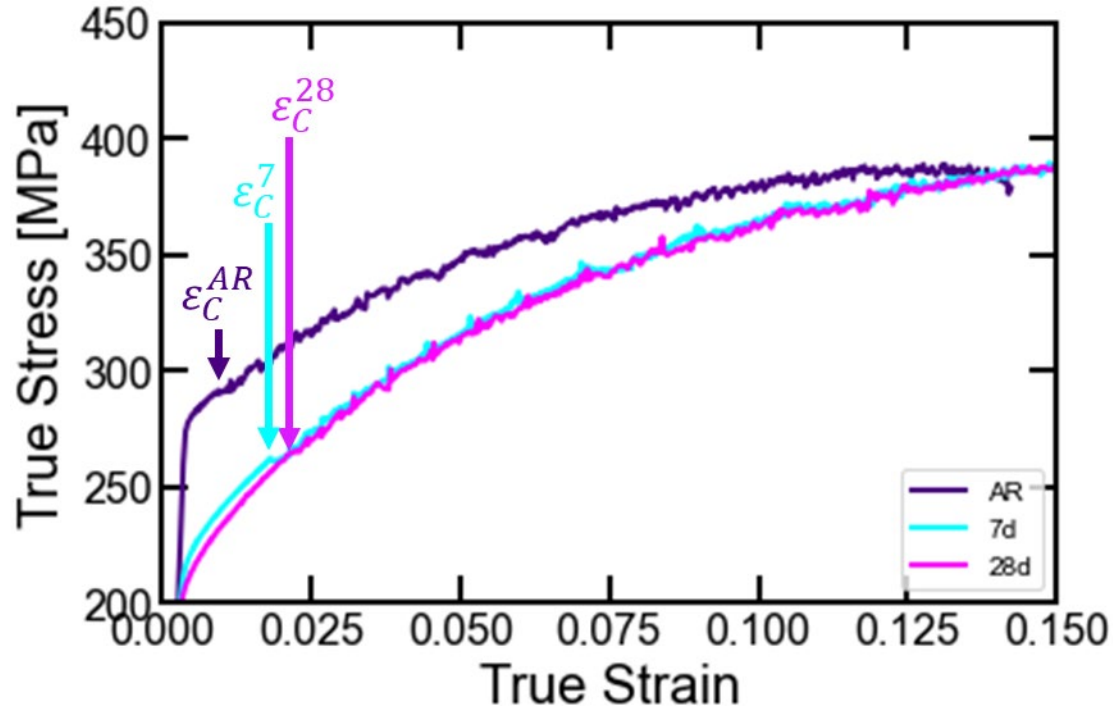


Fig. 19 Stress-strain curves of as-received and sensitized Ω -5083-H131 highlighting the critical strain and the serrated flow from DSA

The increased critical strain is more likely a consequence of the long-term annealing reducing the dislocation density. While the effect of dislocation density on the onset of PLC effect has not been explicitly studied, several investigations examined how cold work or annealing altered the localization of strain in Al-Mg alloys.^{47,50} In general, as the dislocation density is decreased, the critical strain for the onset of the PLC effect increases; the magnitude of this effect is found to be

much greater than the effect of Mg solute leaving the matrix (the existing dislocation network is more impactful on mechanisms than the solute content).³⁷

The specific contributions of the change in Mg content compared to that of the change in dislocation density requires further detailed study to elucidate the mechanisms; what is clear from the current observations of the stress–strain curves is that the interaction of Mg and dislocations changes as a function of sensitization. However, this is primarily an influence on the strain hardening response, rather than the yield strength; in Zhou et al.⁴⁹ it was shown that the changes in DSA did not affect the solid-solution strengthening mechanism for yield strength.

3.3.2 Dislocations

In order to describe the primary way dislocations contribute to the yield strength, Huskins et al. chose the dislocation wall mechanism based on their TEM observations:⁸

$$\sigma_{DW} = \tilde{k}l^{-1/2} \quad (8)$$

In addition to the wall substructures, dislocation substructures can also take the form of tangles or other networks.³⁸ In the work of Goswami and Holtz in 5083-H116, dislocations began as tangles, then evolved to wall substructures after sensitization aging at 100 °C.⁴² In our TEM investigation of Ω -5083-H131 plate, the dislocation substructures appeared as tangles in the as-received condition, as seen in Fig. 20A at two magnifications. After 28 days at 100 °C, the dominant dislocation structure still appears to be tangles, Fig. 20B. However, some evidence of dislocation wall substructures can also be found, Fig. 20B upper right and Fig. 20C. The as-rolled dislocation substructure and its evolution during sensitization is another source of variability both within a given plate and between plates from different suppliers.

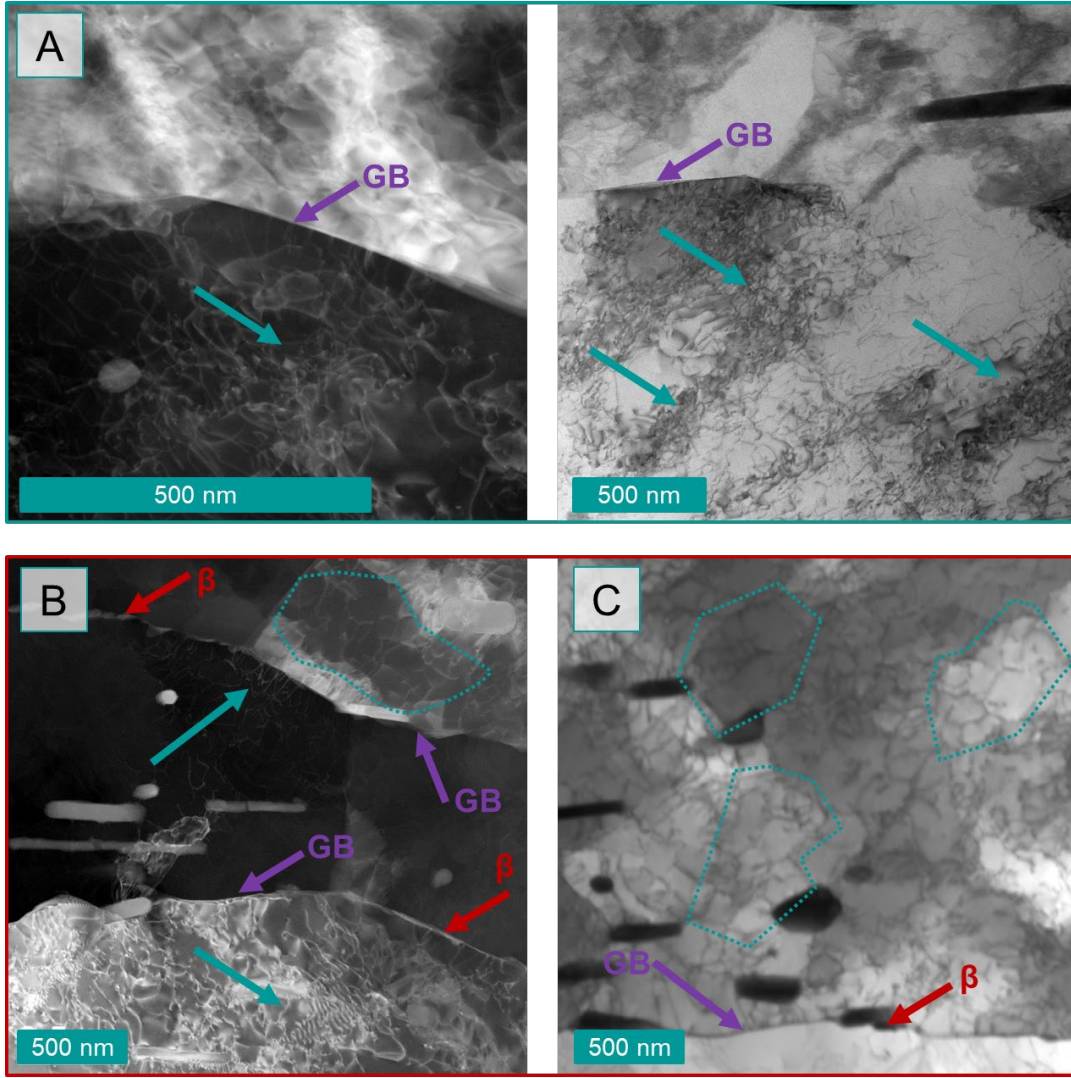


Fig. 20 TEM image of dislocation tangles in (A) as-received condition; (B) 28 days at 100 °C sensitized condition with mostly dislocation tangles (teal arrow) and some wall substructures (dotted region); and (C) 28 days at 100 °C sensitized condition for the Ω -5083-H131 plate with mostly dislocation wall substructures

The contribution of dislocation tangles to yield can be described by

$$\sigma_{DD} = M\alpha Gb\sqrt{\rho_0} \quad (9)$$

Where M is the Taylor factor (~ 3 for aluminum alloys), α is a material constant, G is the shear modulus and b is the Burgers vector.

However, what is more important in the current study is the rate of change of dislocation density with temperature exposure. An interesting approach to time-dependent dislocation recovery and recrystallization in 5083-H116 by Summers et al.⁵¹ included rate dependent evolution of dislocation sub-walls, including

comparison to some experimental evolution of wall spacing at 120 °C. In the referenced Xing et al.,⁵² the observed spacing change from 400 to 470 nm after only 2 h at 120 °C would correlate to a drop in yield stress of approximately 7.8 MPa. As the work of Summers et al.⁵¹ is based on sub-walls rather than tangles and is mostly based on higher temperature data (e.g., >200 °C) we turn to other models in this study. However, this model may be useful as a basis for future modeling of cyclic temperature profiles due to the inclusion of temperature rate effects, which is novel compared to previous recovery/recrystallization models.

The work of Nes³⁸ includes multiple mechanisms for dislocation recovery and also data for both aluminum at 100 °C and multiple Al-Mg compositions at room temperature. In particular, he compares the different mechanisms for Al-4wt% Mg, of direct relevance to AA5083. The experimental data on the change in yield strength with time, $\sigma(t)$, due to dislocation recovery can be well modeled with the following form:

$$\sigma(t) = \sigma_0 - \frac{1.5\sqrt{\rho_0}}{\alpha_3\kappa_2b^2} \ln\left(1 + \frac{1}{\tau}\right) \quad (10)$$

Where σ_0 is the initial yield strength, and τ is a time constant:

$$\tau = \left[\frac{\sqrt{\rho_0}A_1B_x}{kT} \exp\left(\frac{A_1}{kT}\right) \right]^{-1} \quad (11)$$

And A_1 is

$$A_1 = \alpha_3\kappa_2Gb^3 \quad (12)$$

The remaining terms in these equations are described in Table 13. There are multiple possible mechanisms for dislocation recovery,³⁸ the one referenced and used herein is the mechanism determined to be appropriate for Al-Mg at room temperature and pure Al low temperature by Nes. We use the coefficients determined by Nes for Al-4Mg and others referenced in Table 13.

Table 13 Dislocation recovery model parameters

Parameter	Value	Unit	Description
ρ_0	1.4×10^{14}	$1/\text{m}^2$	initial dislocation density (H18) ³⁸
$\alpha_3 K_2$	0.14	...	fitted constant ³⁸
τ	0.12	s	fitted time constant ³⁸
b	2.88×10^{-10}	m	burgers vector ⁸
G	27000	MPa	shear modulus ⁸
k	1.380649×10^{-23}	J / K	Boltzmann constant
T	25–100	°C	temperature
	298–373	K	

Some estimates for the drop in yield strength due to dislocation recovery using the Nes model are shown in Fig. 21. Figure 21A shows the difference in yield drop from dislocation recovery for the sensitization temperatures of 70 and 100 °C. This prediction uses a starting dislocation density, ρ_0 , of 1×10^{14} and a Mg concentration of 4 wt% and shows that the yield drop increases with increasing sensitization temperatures. Figure 21B shows the difference between a low Mg/low dislocation density starting material compared to a high Mg/high dislocation density starting material. These values were chosen to represent the likely lower and upper bounds of dislocation recovery in 5083-H131, that is, the minimum and maximum allowable Mg contents from MIL-DTL-46027² and likely dislocation densities for the cold worked H131 temper that were estimated from literature. Yi et al.¹⁷ measured the geometrically necessary dislocation (GND) density in a plate of 5083-H131 at $5.33 \times 10^{13} \text{ m}^{-2}$; barring better experimental measurements, the GND density can be assumed to be about half of the total dislocation density for a total dislocation density of approximately $1 \times 10^{14} \text{ m}^{-2}$. Converting the measurements of Huskins et al.,⁸ an estimated dislocation density would be approximately 0.9×10^{14} for their 5083-H131 plate. The dislocation density measurement of 2.2×10^{14} in Nes³⁸ was for a plate of H18 temper, which should be higher than H131 as it receives more cold work.

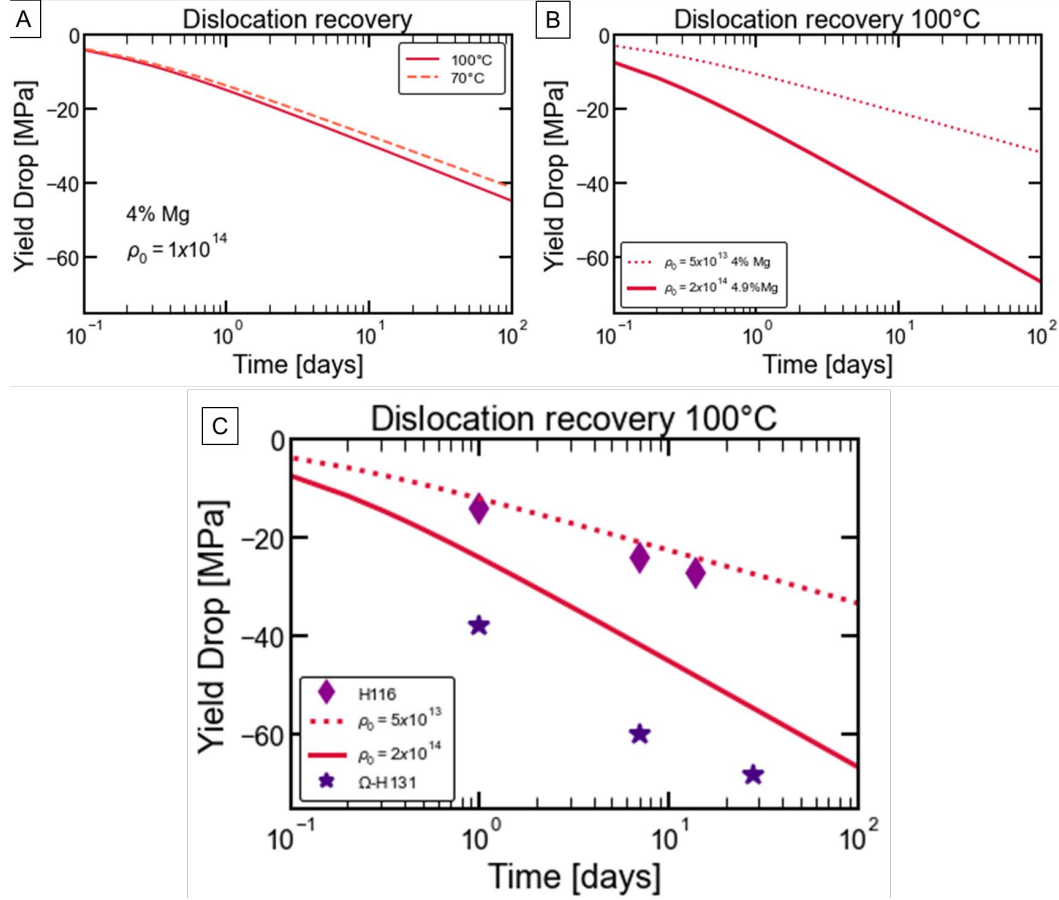


Fig. 21 Predicted drop in yield strength due to dislocation recovery using the model of Nes and the parameters in Table 13

The estimates of Fig. 21 are particularly useful in explaining the observed difference in yield drop with sensitization level between the H131 and H116 tempers shown in Fig. 14. Despite similar values for DoS, the yield drop in the H116 is less than half that of H131. With a much lower starting dislocation density in H116, that is, the dotted line in Fig. 21C compared to the solid line, which approximates H131, the yield drop at a given temperature and exposure will always be considerably lower. The measured yield drop at 100 °C for the H116 plate and the Ω-H131 plate are plotted in Fig. 21C compared to the estimate of dislocation recovery at the same temperature. The starting dislocation densities are estimated from literature, with measured GNDs of approximately 5×10^{13} from 5083-H116.³⁵ The difference between the dislocation-associated strength decrease and the actual observed drop is due to the evolution of the magnesium solid solution strengthening drop, as discussed in the next section.

3.3.3 Total Yield Drop Discussion

Given the previous discussion of the likely sources of the drop in the yield strength in sensitized 5083, we summarize and compare to the measured values in the Ω -5083-H131 plate. The absolute values of the yield drop in this plate in the sensitized conditions were -38 , -60 , and -68 MPa for 1, 7, and 28 days at 100°C .

The minimum and maximum predictions from the various sources of yield drop are shown in Table 14 for a 28-day at 100°C sensitized condition. The maximum value for the contribution of β on grain boundaries is calculated by assuming the entire measured grain boundary area has nucleated β phase. Combining the contributions, the predicted yield drop is 31 to 75 MPa. The observed drop of 68 MPa agrees well with the total predicted decrease in strength.

Table 14 Summary yield drop (for Ω -H131 plate 28 days)

Source		Max value (MPa)	Min value (Mpa)	Description
Dislocation Recovery		54.62	25.66	Section 3.3.2
Mg / solid solution strengthening	β GB	10.99	5.455	Section 3.3.1.1, Table 11
	β on Al_6Mn	9.367	0.1877	Section 3.3.1.2, Table 12
	Mg on dislocations	NA	NA	Section 3.3.1.3

From the preceding analysis, it appears that the dislocation recovery due to elevated temperature exposure is a more important factor than the loss of Mg from solid solution during the formation of β phase. However, the predictions of Table 14 for the Mg mechanisms are based directly on the measured microstructural features of the Ω -5083-H131 plate. The highest GB coverage we observed was 55% of the GB covered by the β phase. Other works have observed higher coverages, mostly for other tempers. In Jain et al.,⁵³ a 5083-H131 plate with a similar DoS to our 55% GB coverage observation saw about 66% of the GB coverage, also sensitized at 100°C . At a higher DoS the coverage was 76%. In H116 temper, up to 81% of the GB was covered in β ,³⁴ a solutionized 5083 saw up to 80% coverage,⁵³ and there was up to 90% coverage in an H128 sample, but this was observed at a sensitization temperature of 175°C , which will have significantly faster kinetics.¹⁸ If we assume that the GB can be completely saturated with β phase (which has been observed in higher temperature sensitization but not yet for lower temperature), the amount of Mg pulled from solid solution could be on the order of 1 wt%, resulting in a drop of approximately 15 MPa from the yield strength. This is still less than the anticipated yield drop from dislocation recovery. The relative expected changes due to sensitization are shown in Fig. 22.

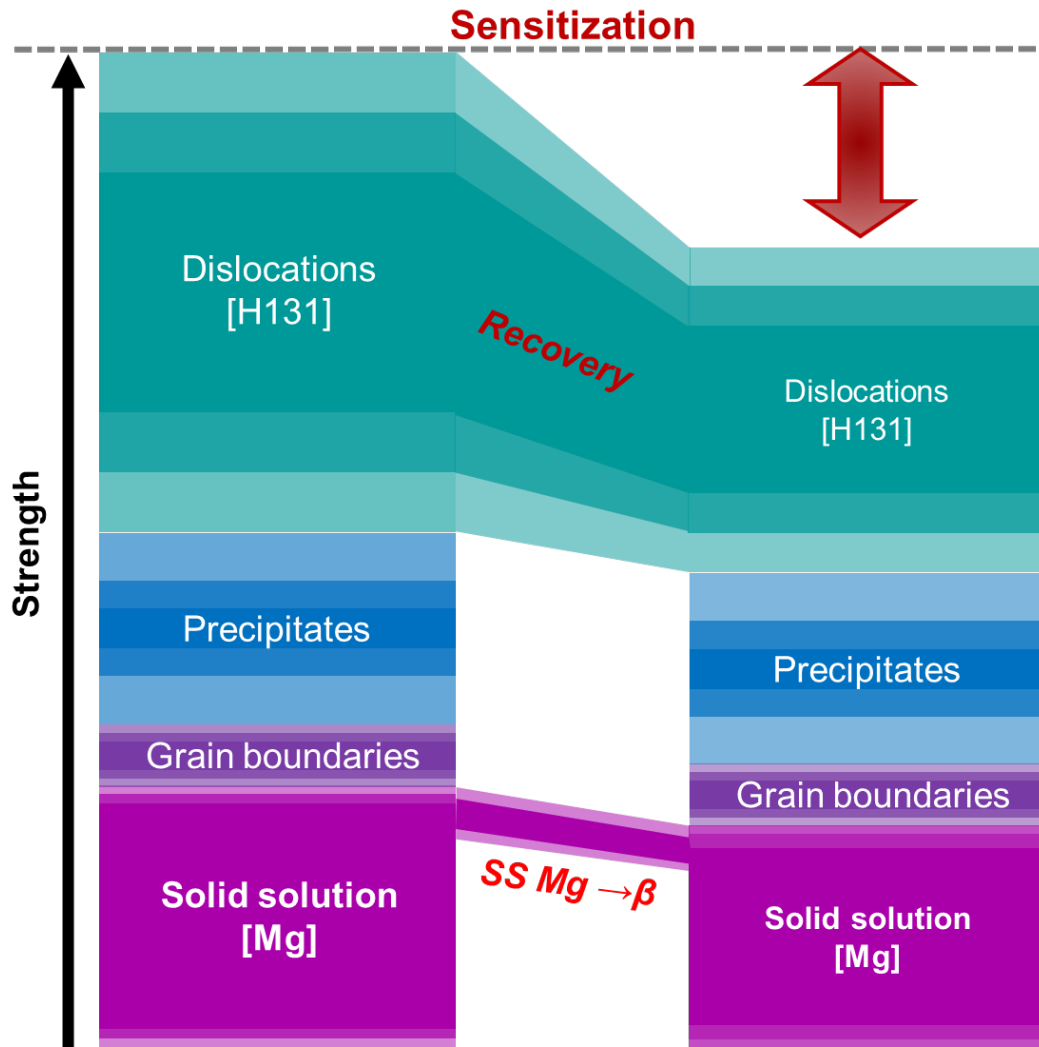


Fig. 22 Schematic of the relative change in strengthening mechanisms. Gradient colored bands represent the possible variability in these values between plates.

One proposed solution for sensitization is thermal remediation, which redissolves the β -phase and returns Mg to solid solution. However, this approach will not recover the dislocation density and its corresponding contribution to strength. In fact, in Seifi et al.⁵⁴ the yield strength was not observed to recover considerably after remediation treatment; following a sensitization-induced strength decrease of 65 MPa, only between 6 and 14 MPa in strength was recovered after the various remediation conditions. In 5456,⁵⁵ thermal remediation also did not recover the yield strength in any substantial way. Further, given the short times involved for the remediation treatment, it is likely that only a localized redissolution of Mg occurs and not a complete recovery of the full solid solution strengthening. Specifically, insufficient time is available for Mg to diffuse through the Al matrix.

The limited efficacy of the remediation treatments in recovering the strength is unsurprising as they were proposed and developed with the intent of reducing the sensitization-related corrosion issues. Such efforts were successful at decreasing the locally measured DoS, which would decrease susceptibility to IGC. However, as indicated by both their results and the now more complete understanding of the mechanisms of yield strength drop in sensitized material developed here, it is not a viable solution for strength remediation. The measurements of Seifi et al.⁵⁴ support the relative contributions of the loss from Mg solid solution strengthening and the loss from dislocation recovery to the yield drop estimated herein.

4. Conclusions

Multiple plates of 5083-H131, representing a variety of suppliers and plate thickness, were assessed for their susceptibility to sensitization. More variability in the degree of sensitization was observed at the lower sensitization temperature (70 °C). The H131 behavior was also compared to an example 5083-H116 plate that was known to be highly susceptible to sensitization. All sensitized plates and conditions exhibited a decrease in the yield strength. Microstructural characterization was performed on one plate in conjunction with a detailed examination of strengthening model parameters in order to establish the specific mechanisms contributing to the yield drop.

The yield drop in sensitized 5083 is caused by both Mg leaving solid solution to form the β phase and by dislocation recovery. The proportional effect of these two mechanisms will vary depending on plate (e.g., its initial dislocation density, grain boundary density and character, initial precipitate density, and overall Mg content), but it is generally expected to be influenced more by the dislocation recovery.

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List of Symbols, Abbreviations, and Acronyms

APT	atom probe tomography
ATC	Aberdeen Test Center
DIC	digital image correlation
DoS	Degree of Sensitization
DSA	dynamic strain aging
EBSD	electron backscattered diffraction
EDS	energy-dispersive X-ray spectroscopy
GBs	grain boundaries
GND	geometrically necessary dislocation
JC	Johnson–Cook
JMAK	Johnson–Mehl–Avrami–Kolmogorov
NAMLT	Nitric Acid Mass Loss Test
PLC	Portevé–Le Chatelier effect
SEM	scanning electron microscopy
TEM	transmission electron microscopy
UTS	ultimate tensile strength
UTM	universal testing machine

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1 DOD CPO
(PDF) R HERRON

13 DEVCOM ARL
(PDF) FCDD RLA M
B PLACZANKIS
B CHEESEMAN
FCDD RLA MB
D MAGAGNOSC
T WALTER
D O'BRIEN
FCDD RLA MC
L BLOHM
J KELLEY
J SNYDER
FCDD RLA MF
H MURDOCH
T CAIN
K DOHERTY
M WALOCK
FCDD RLA TE
D GALLARDY