Mitigating Abnormal Grain Growth for Friction Stir Welded Al-Li 2195 Spun Formed Domes

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Abstract

Formability and abnormal grain growth (AGG) are the two major issues that have been encountered for Al alloy spun formed dome development using friction stir welded blanks. Material properties that have significant influence on the formability include forming range and strain hardening exponent. In this study, tensile tests were performed for two 2195 friction stir weld parameter sets at 400 °F to study the effects of post weld anneal on the forming range and strain hardening exponent. It was found that the formability can be enhanced by applying a newly developed post weld anneal to heat treat the friction stir welded panels. This new post weld anneal leads to a higher forming range and much improved strain hardening exponent. AGG in the weld nugget is known to cause a significant reduction of ductility and fracture toughness. This study also investigated how AGG may be influenced by the heating rate to the solution heat treatment temperature. After post-weld annealing, friction stir welds were strained to 15% and 39% by compression at 400 °F before they were subjected to SHT at 950 °F for 1 hour. Salt bath SHT is very effective in reducing the grain size as it helps arrest the onset of AGG and promote normal recrystallization and grain growth. However, heat treating a 18 ft dome using a salt bath is not practical. Efforts are continuing at Marshall Space Flight Center to identify the welding parameters and heat treating parameters that can help mitigate the AGG in the friction stir welds.

I. Introduction

Cryogenic fuel tanks used in rocket vehicles are of diameters in the range of 12 feet or greater. Large rocket cryogenic tank domes have typically been fabricated using Al-Cu based alloys like Al-Cu alloy 2219. The use of aluminum-lithium based alloys can reduce weight because aluminum-lithium alloys have lower density and higher strength than Al-Cu alloy 2219. However, Al-Li alloys have rarely been used to fabricate fuel tank domes because of the inherent low formability characteristic that make them susceptible to cracking during the forming operations. Joining Al-Li alloys with fusion welding adds to the complexity and cost of cryogenic tank fabrication also because of the susceptibility to cracking and defect formation during welding. Friction stir welding (FSW) as a solid state joining process has proven to be a perfect solution for manufacturing cryogenic tanks with these alloys.

NASA is interested in developing the technology that would enable a single piece, net-shaped dome for cryogenic fuel tanks from Al-Li 2195 alloy using spin forming. This application requires large plates with diameters greater than 18 ft. Commercial plate is not available in sizes large enough to fulfill this requirement, but large blanks can be fabricated by joining smaller plates with FSW.

Several technical challenges must be overcome when spin forming Al-Li alloy 2195 using friction stir welded blanks. First, the ability to form metal by spin forming without excessive thinning or necking depends on the strain hardening exponent “n”. A high strain hardening exponent is beneficial to a material’s ability to uniformly distribute the imposed strain. Also, FSWs that have undergone solution heat treatment (SHT) can develop abnormal grain growth (AGG) in the nugget zones, which significantly reduces the mechanical properties. AGG can also arise during post-weld annealing and is sensitive to plastic deformation of the weld.

The as-welded blank is not suitable for spin forming because there is an unequal hardness distribution in the weld nugget, the thermo-mechanically affected zone, the heat affected zone and the base material. This leads to an inhomogeneous forming behavior and uneven material thinning between the nugget and the base material. Therefore the as-welded blank must be heat treated prior to spin forming.

To spin form an aluminum lithium alloy dome blank joined by FSW, the hardness distribution across the spin blank must be equalized by a heat treatment called “post weld anneal”. More importantly, the formability for such blanks should not be adversely affected by this post weld annealing treatment. MSFC has developed a novel annealing process that can achieve a work hardening exponent on the order of 0.27 to 0.29, which is approximately 50% higher than what is typically obtained for Al-Li alloys using the conventional method.

AGG mitigation for the spun-formed 2195 dome is complicated because the forming degree can vary between 5 to 40% along the dome axis. Another main objective of this study is to identify a post forming heat treatment method that can mitigate AGG and improve the mechanical properties. The AGG mitigation efforts for FSW 2195 is continuing at Marshall Space Flight Center and the results of the present study will be presented.

II. Experimental Procedures

1. Material and Friction Stir Welding

The alloy selected for this study is AA2195 alloy that belongs to the Weldlite (Al-Cu-Li) family. The nominal composition of AA2195 is shown in Table 1. AA2195 is one of the most complicated alloys due to the precipitation of a large number of second phase particles. Details of the microstructure evolution and precipitation behavior are published elsewhere [1-3].

Table 1. Nominal chemical composition of Al-Li 2195

Alloy	Cu	Li	Ag	Mg	Zr	Others
2195	4.0	1.0	0.3	0.35	0.10	<0.05 each

Conventional friction stir welding was used to join the plates creating the spin forming blank. That is, the applied forging load was reacted by a steel anvil. Weld parameters were chosen to achieve a desired grain size in the nugget, rather than to optimize mechanical properties. Previous work indicated that larger grains in the nugget of conventional friction stir welds were less susceptible to abnormal grain growth after subsequent processing operations than small grains. The pin tool design was a standard configuration used at MSFC. An optimized weld nugget shape was not evaluated as part of this effort, although it is believed to have an effect on subsequent spun formed mechanical properties. The C-FSW butt welds were produced with the welding direction parallel to the plate rolling direction. Two welds, weld 1 and MT weld were used for the formability study. Weld 1 was produced at Marshall Space Flight Center and MT weld was furnished by MT Aerospace. Weld nugget grain size was determined using electron backscatter diffraction (EBSD) analysis using a FEI Quanta 600F field emission scanning electron microscope. The grain size is approximately 7.0 µm for weld 1 and 10.0 µm for MT weld.

2. **Heat treatment for formability study and microhardness testing**

After welding, all welds were annealed using three annealing treatments (1) PW anneal 1; (2) PW anneal 2; (3) MT anneal. PW anneal 2 is a new formability enhancing heat treatment method developed at NASA-Marshall Space Flight Center [4]. According to this method, the product is first heated to a temperature within the range of 204° to 343° C. (400° to 650° F), and held at the temperature reached for up to 16 hours. Thereafter, the product is subjected to a controlled heating process at a heating rate of 3° C/hour or higher to a second temperature within the range of 371° to 482° C. (700° to 900° F), held at the temperature reached for up to 12 hours. Then the product is subjected to a controlled cooling process at a cooling rate of 3° C/hour or higher to a third temperature within the range of 204° to 343° C. (400° to 650° F), and held at the temperature reached for up to 4 hours. Then the product is cooled to room temperature. Figure 1 graphically depicts the heat treating process of PW anneal 2. The schedule for MT anneal is proprietary.

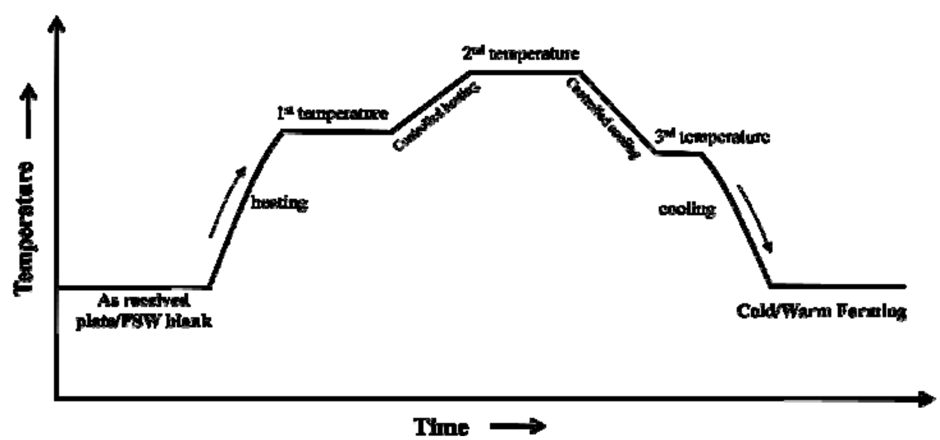

Figure 1. A schematic graph illustrating the heat treating process of PW anneal 2.

The weld hardness was measured using a microhardness tester to examine the local variations in the hardness in and around the weld zone before and after the post weld anneal. The microhardness tests were performed at room temperature, approximately 72 °F.

3. **Tensile Testing**

Tensile testing was performed to determine the mechanical properties and strain hardening exponent. Tensile specimens were taken along the T direction from the welded plates. Specimens with a double reduced gage section were used to ensure that the specimen would neck and break in the nugget of the weld. The specimen configuration is shown in Figure 2. Tensile test were performed at 200° C (396 °F) with a strain rate of 0.03 in/in/minute. The weld nugget is completely inside the 0.5” gauge section. A 0.3” extensometer was used to record the strain within the 0.5” gage section of the specimen.

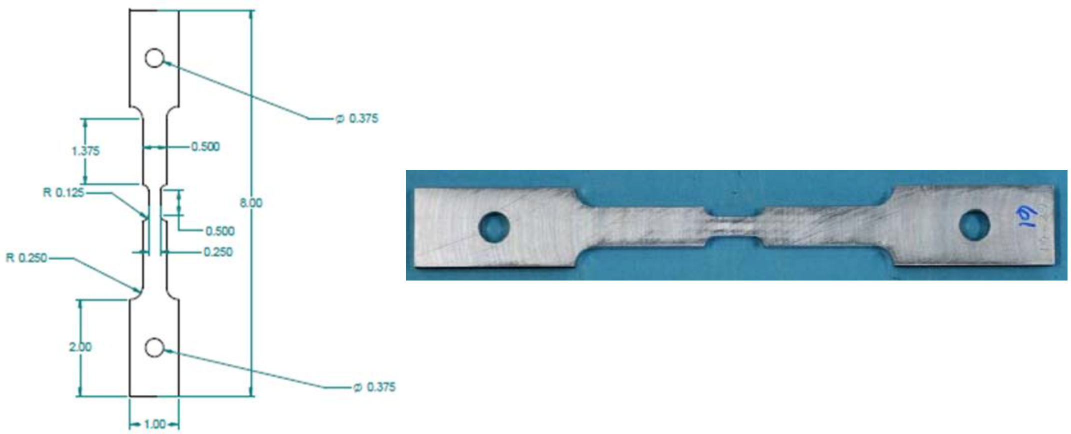

Figure 2. The tensile specimen with a double reduced gage section. The weld nugget is completely inside the 0.5” gauge section.

The value of the strain hardening exponent (n) was determined using true stress-true strain diagram by Eq. 1.

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$$\sigma = K\varepsilon^{n} \qquad n = \left(\frac{\partial \ln \sigma}{\partial \ln \varepsilon}\right)_{\dot{\varepsilon}} \qquad \text{(Eq.1)}$$

The tensile test matrix for weld 1 and MT weld is shown in Table 2 and 3, respectively.

Table 2. The tensile test matrix for weld 1

Weld 1			
Post Weld Anneal	Testing Temperature	Specimen location	Orientation

PW Anneal 1	200° C (396 °F)	weld	LT
PW Anneal 2	200° C (396 °F)	weld	LT

Table 3. The tensile test matrix for MT weld

MT Weld			
Post Weld Anneal	Testing Temperature	Specimen location	Orientation
MT Anneal	200° C (396 °F)	weld	LT
PW Anneal 2	200° C (396 °F)	weld	LT

4. Compression Straining at 200°C (396°F)

To simulate the forming strains incurred during spin forming, the C-FSWs were subjected to compression straining. Cylindrical compression specimens measuring 1 in.-tall x 0.375 in.-diameter were machined from the C-FSW panel with the specimen axis oriented transverse to the plate rolling direction with the weld seam bisecting the specimen length (see Figure 3). Compression tests were performed at 200 °C (396 °F) in air with a strain rate of 0.03 in/in/minute. The cylindrical specimens were axially loaded in compression to various strain level increments as detailed in the test matrix shown in Table 4. The total deformation strain of 39% approximates the maximum forming degree in the dome membrane region.

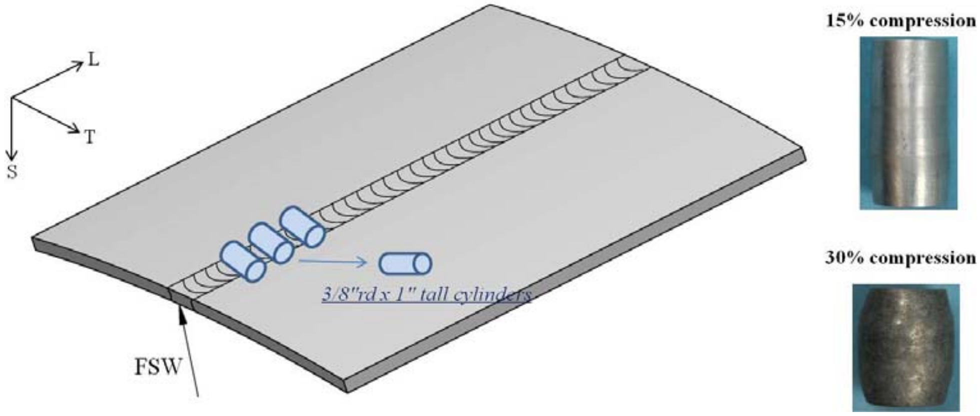


Figure 1. Schematic of FSW blank showing orientation of the compression specimens.

5. Anneal and Solution Heat Treatment

Following the compression straining, the specimens were annealed and solution heat treated to study the microstructural evolution of the weld. To investigate the effects of heating rate to the SHT temperature on AGG, three different anneal/SHT schedules were used as shown in Table 4 and Table 5. One of these schedules, anneal/SHT 1, was conducted in a conventional air furnace, while the other two schedules, anneal/SHT 2 and anneal/SHT 3, were conducted in a salt bath. Following solution heat treatment, the weld specimens were sectioned, mounted, polished and etched for metallurgical examination.

Table 1. Compression deformation and heat treatment test matrix.

Weld	1st Strain Increment	Anneal	2nd Strain Increment	Anneal	3rd Strain Increment	Total Strain	Heat Treatment
Weld 1	15%	no	-	no	-	15%	Anneal/SHT 1
Weld 1	15%	no	-	no	-	15%	Anneal/SHT 2
Weld 1	15%	no	-	no	-	15%	Anneal/SHT 3
Weld 1	15%	no	15%	no	15%	39%	Anneal/SHT 1
Weld 1	15%	no	15%	no	15%	39%	Anneal/SHT 2
Weld 1	15%	no	15%	no	15%	39%	Anneal/SHT 3

Table 5. The heat treatment schedule used for compression strained specimens.

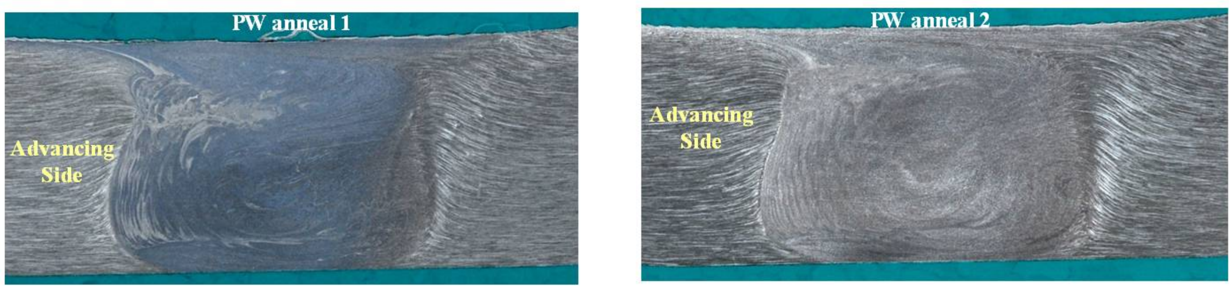
Heat Treatment	Schedule
Anneal/SHT 1	R/C heating to 800°F + 800°F/4h + R/C heating to 950°F + 950°F/1h + WQ
Anneal/SHT 2	Immersion in a 950°F salt bath + 950°F/1h + WQ
Anneal/SHT 3	R/C heating to 800°F + 800°F/4h + immersion in a 950°F salt bath + 950°F/1h + WQ

R/C – ramp controlled
A/C – air cooling

III. Results and Discussion

1. Nugget zone microstructure

Macroscopic views of the nugget zone after post weld anneal for weld 1 and MT weld are shown in Figure 4 and 5, respectively. The nugget shape of weld 1 clearly shows the weld parameters used resulted in a non optimized nugget. Although bulging is readily seen, internal voids did not develop. The typical onion ring structure can be clearly seen in weld 1 after post weld anneal. The nugget zone is not clear after MT anneal, but was revealed nicely after PW anneal 2. This finding suggests that the temperature for MT anneal is lower than that for PW anneal 2.



(a) (b)

Fig. 4. Macro images of the nugget zones for weld 1 after (a) PW anneal 1 and (b) PW anneal 2.

(a) (b)

Fig. 5. Macro images of the nugget zones for MT weld after (a) MT anneal and (b) PW anneal 2.

2. Microhardness

Microhardness testing was conducted to examine the local variations in the hardness around the friction stir weld zone. The microhardness profile around the weld for weld 1 and MT weld is shown in Figure 6. MT weld with MT anneal has the highest hardness in the weld nugget and shown an unequal hardness distribution in the weld nugget and base metal. The weld/parent metal hardness was more equalized using PW anneal 1 and PW anneal 2 for both weld 1 and MT weld. This finding indicates that the temperature for MT anneal is lower than that for PW anneal 1 and PW anneal 2.

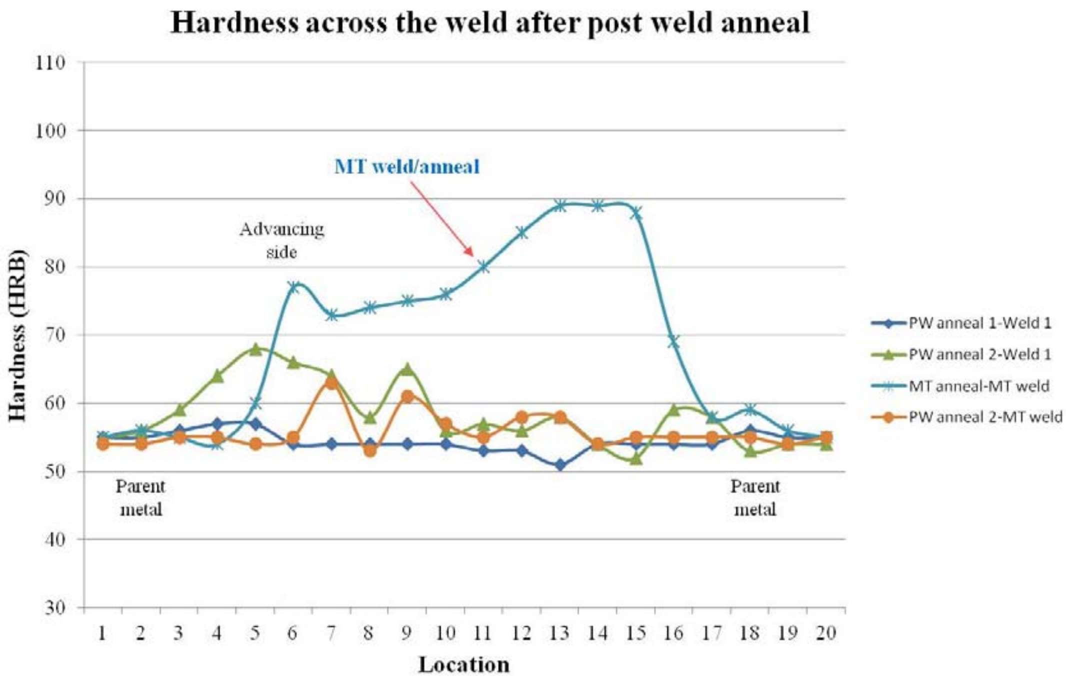


Fig. 6. Comparison of hardness profile across the weld for Weld 1 and MT weld after various post weld anneals.

In general, the welded blank is not suitable for forming operations when there is an unequal hardness distribution in the weld nugget, the thermo-mechanically affected zone, and the heat affected zone and the base material. The unequal hardness distribution would result in an inhomogeneous forming behavior and uneven material thinning between the nugget and the base material. Therefore the as-welded blank must be annealed prior to spin forming. A more homogenous forming behavior in the welding area can be achieved with the use of PW anneal 1 and PW anneal 2, although there is still a small variation in microhardness across the weld nugget.

3. Tensile Properties and Forming Range

The effects of post weld anneal on the tensile properties are shown in Table 6. In general, a lower value of yield strength and a higher value of tensile strength gives greater formability. Therefore, the forming range, which is the difference between the tensile strength and yield strength, was used to compare the formability. In most cases a higher value of forming range would be expected to lead to a greater formability [5].

Table 6. Tensile properties of weld 1 and MT weld with different post weld anneals.

Material	Anneal	Yield Stress (ksi)	Tensile Stress (ksi)	Ductility (%)	*Forming Range (ksi)	Formability Ranking
Weld 1	PW anneal 1	11.5	16.6	65.9	5.1	***3***

Weld 1	PW anneal 2	9.3	18	63.3	8.7	***1***
MT Weld	MT anneal	17.7	22	58.2	4.2	***4***
MT Weld	PW anneal 2	11.5	20	60.8	8.6	***2***

*: forming range denotes tensile strength - yield strength

For weld 1, PW anneal 2 is the better heat treatment than PW anneal 1 for forming operations as it leads to a lower yield strength and a higher ultimate tensile strength. Accordingly, the forming range increases from 5.1 ksi (by PW anneal 1) to 8.1 ksi by the use of PW anneal 2, a more than 60% improvement.

PW anneal 2 is also superior to MT anneal for MT weld. The forming range for MT weld increases from 4.2 ksi (by MT anneal) to 8.6 ksi with the use of PW anneal 2, a more than 80% improvement. The overall formability ranking based on the forming range is shown in Table 6.

4. **Strain Hardening Exponent (n)**

Figures 7 is an illustration of true stress *vs.* true strain curves for weld 1 and MT weld after three post weld anneals. It can be seen that the annealing schedule has profound effects on the tensile behavior. MT anneal for MT weld gives high yield strength but accelerated strain softening, which are signs of poor formability. Anneal 2 is the most effective in increasing the strain hardening for both MT weld and weld 1**.**

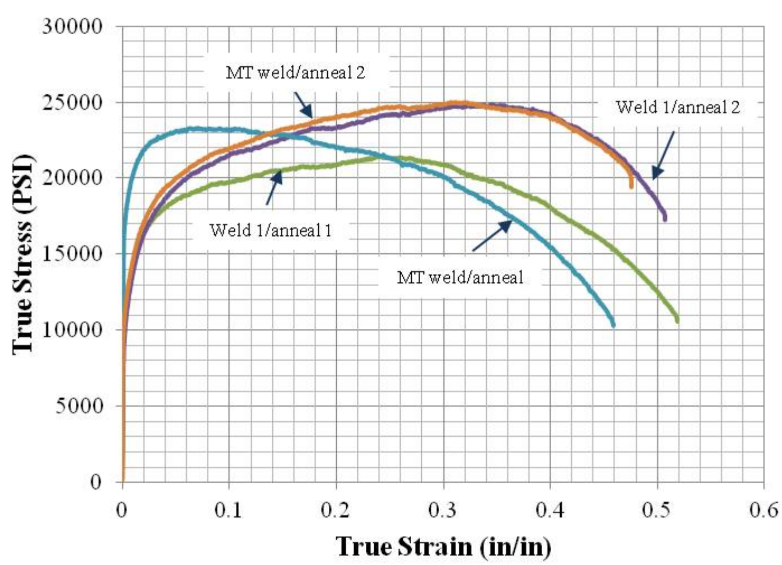


Fig. 7. The true stress-true strain curves for Weld 1 and MT weld after different anneals.

The strain hardening exponent, n, which indicates the ability of the material to distribute the strain uniformly prior to diffuse necking, was calculated for all tensile tested samples using Eq. 1. A material with a higher value of n indicates more uniform strain distribution and hence greater formability [6].

It can be seen from Table 7 that the strain hardening exponent is higher when annealed using PW anneal 2 for both weld 1 and MT anneal. With the use of PW anneal 2, the strain hardening exponent for weld 1 was increased from 0.16 to 0.21 in the 1% to 5% strain range, and from 0.1 to 0.15 in the 5% to 9% strain range.

Table 7. Strain hardening exponent of weld 1 and weld 2 with different post weld anneals.

Material	Anneal	Strain Hardening, n (1 to 5% strain)	Strain Hardening, n (5 to 9% strain)	Formability Ranking
Weld 1	PW anneal 1	0.16	0.1	***3***
Weld 1	PW anneal 2	0.21	0.15	***1***
MT Weld	MT anneal	0.08	0.01	***4***
MT Weld	PW anneal 2	0.19	0.15	***2***

*: forming range denotes tensile strength - yield strength

PW anneal 2 also works very well for MT weld. The strain hardening exponent after MT anneal was only 0.08 in the 1% to 5% strain range, and dropped to 0.01 in the 5% to 9% strain range. By applying PW anneal 2, the strain hardening exponent was drastically increased from 0.08 to 0.19 in the 1% to 5% strain range, and from 0.01 to 0.15 in the 5% to 9% strain range.

Based on the strain hardening exponent values, PW anneal 2 is the most promising among the three post weld anneals for 2195 friction stir welds. The formability ranking based on the strain hardening exponent is shown in Table 7. It is significant to note the use of forming range and strain hardening exponent to rank the formability yields the same order. The similarities in strain hardening exponent values for both the weld 1 and MT weld subjected to post weld anneal 2 indicates the nugget grain size used for this study is not a factor in determining the strain hardening exponent.

The strain hardening exponent as a function of true strain for weld 1 and MT weld with different anneals is shown in Figure 8. For MT weld with MT anneal, the strain hardening exponent starts with approximately 0.05 and quickly drops to zero at 7% true strain. A drastic improvement in the strain hardening exponent was observed when the MT weld was treated with anneal 2. The onset of necking was delayed from 3.5% (with MT anneal) to approximately 13.5%. PW anneal 2 also leads to a noticeable increase in the strain hardening exponent for weld 1.

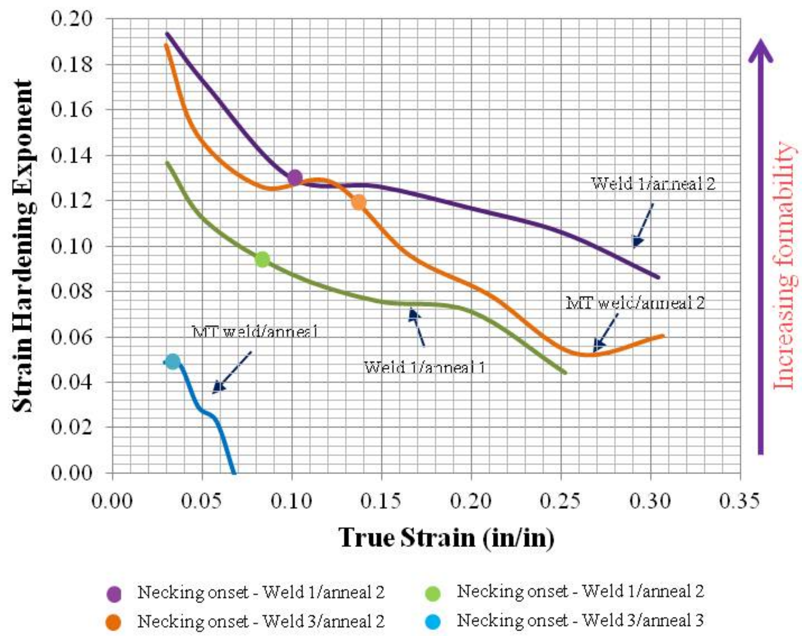
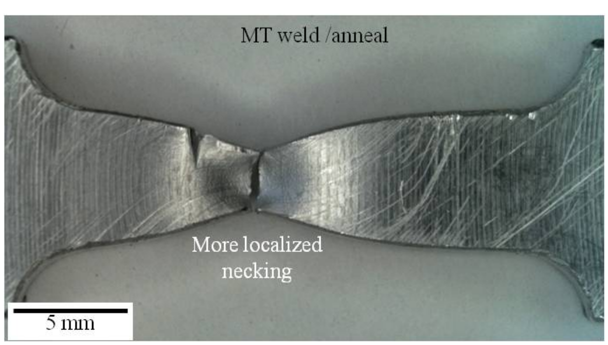
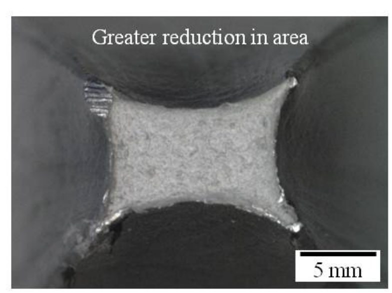


Fig. 8. The strain hardening exponent as a function of true strain for Weld 1 and MT weld after different anneals.

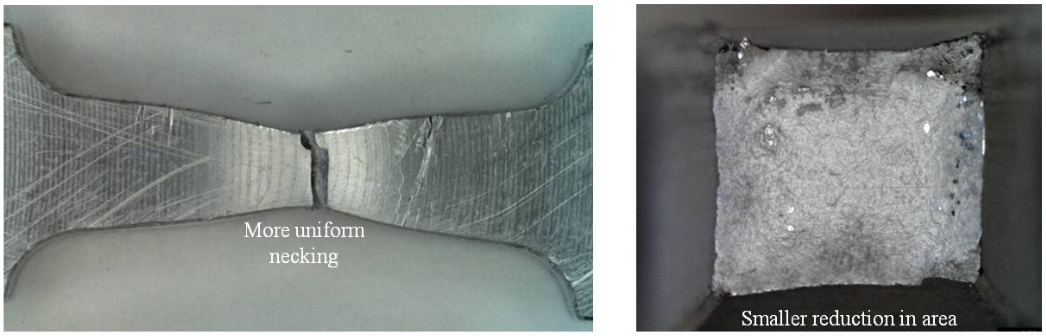
Spin forming is a process during which a blank is clamped and stretch formed well beyond the elastic limit. From the viewpoint of formability in spin forming, a low resistance to thinning (as thinning is inevitable in forming), uniform thinning and low load are important. High-strain uniformity is ensured by a high value of n together with a low YS and high forming range. Forming load would be low with a low YS and high forming range. PW anneal 2 is the most promising post weld anneal as it can lower the yield strength and increase the forming range and strain hardening exponent.

Fractographic analysis of the failed samples is shown in Figure 9. MT weld annealed using MT anneal clearly exhibits more localized necking, a sign of strain localization due to low strain hardening exponent. In contrast, MT weld annealed using anneal 2 exhibits more uniform deformation and diffuse necking, indicative of higher strain hardening exponent. The fractographic observations are in line with the values of strain hardening exponent.





(a)



(b)

Fig. 9. (a) MT weld annealed using MT anneal exhibits more localized necking and the fracture surface has a smaller reduction in area. (b) MT weld annealed using anneal 2 exhibits more uniform necking and the fracture surface has a smaller reduction in area.

5. Abnormal Grain Growth Mitigation

Abnormal grain growth is also called secondary grain growth. According to Humphrey's theory [7], the most effective method of preventing abnormal grain growth of a fine-grained microstructure is with a dispersion of stable second-phase particles. Figure 10 shows the predicted influence of the pinning particles (F_v/d) and the grain size on the stability of fine grain structure. The figure explains the three type of grain growth behavior.

If there are no or few second-phase particles, normal grain growth is predicted to occur during high temperature annealing. However, as F_v/d increases, normal grain growth will be prevented and abnormal grain growth is increasingly likely for smaller grain sizes. Larger grained microstructure is less prone to developing AGG because the microstructure can be stabilized with a lower pinning particle dispersion level.

In 2195, the nugget zone contains both soluble and insoluble second phase particles [2, 3], which can strongly influence the stability of the grain structure by pinning the grain boundaries. For example, Al_3Zr dispersoid particles that are designed to prevent recrystallisation during thermomechanical processing of the parent material will not dissolve during solution treatment.

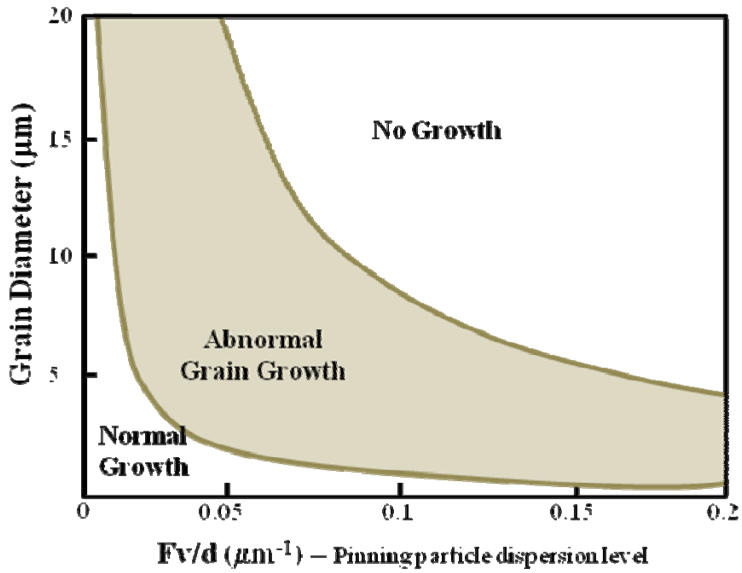


Fig. 10. Predicted regimes of normal, abnormal and no grain growth, as a function of the average grain size and particle pinning pressure (*Fv* /*d*) [1].

T_2 (Al_6CuLi_3), T_B (Al_7CuLi), and θ (Al_2Cu) are soluble constituent phases that will dissolve during SHT [1] and therefore the grain boundary pinning effects will be very low. Based on Humphrey's theory [7], AGG is difficult to stop when the grain boundary pinning particle dispersion level drops rapidly due to the massive dissolution of the second phase particles.

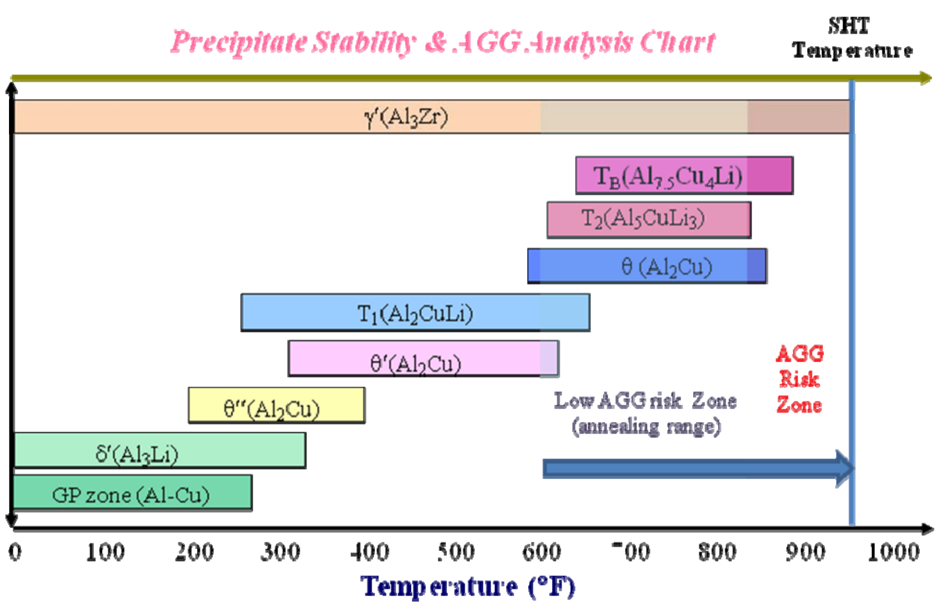


Figure 11. Precipitate stability and AGG risk analysis chart for 2195.

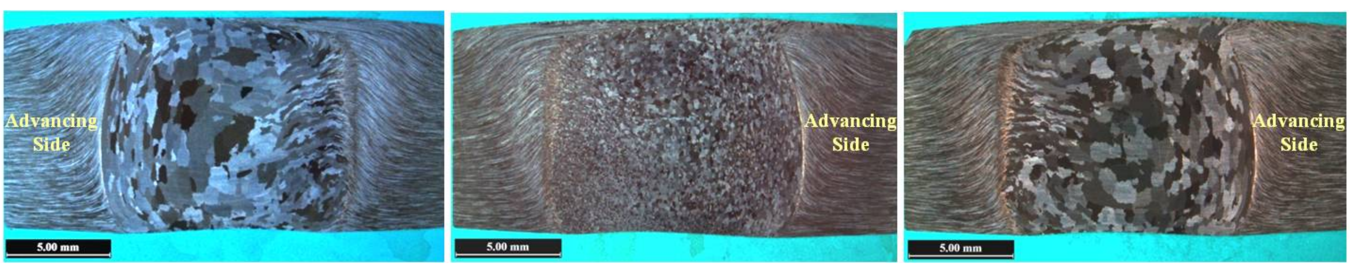
A phase stability chart [3] and AGG risk analysis chart for 2195 is shown in Figure 11 to aid in the understanding of AGG mechanisms and formulate strategies to mitigate AGG. At elevated temperatures (above 800 °F), most of the pinning particles in 2195 become less stable and will progressively dissolve as the temperature continues to rise. So the range of 800 – 950 °F is considered an AGG risk zone due to a drastic reduction in the particle dispersion level.

Based on the AGG risk analysis for 2195, it is thought that AGG can be mitigated by increasing the heating rate through the critical AGG risk temperature range by changing from an air furnace to a salt bath. The heating rate of a salt bath can reach ~500°C/sec, which can be three orders of magnitude faster than the heating rate of a conventional air furnace (10°C/min).

6. Effect of prior deformation strain and heating rate to the SHT temperature (using a salt bath)

Compression specimens were strained to 15% and 39% and heat treated according to the test matrix shown in Table 4. Following the compression straining, the specimens were subjected to anneal/solution heat treatment (see Table 5). Three anneal/SHT schedules were used to investigate the effects of heating rate on the thermal stability of the nugget zone microstructures.

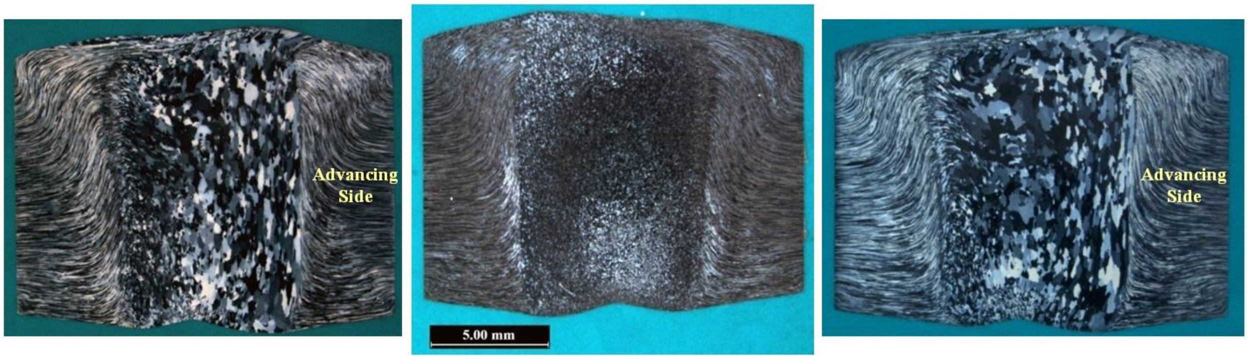
The microstructures of the 15% strained specimens are presented in Figure 12. Rapid heating in a salt bath (anneal/SHT 2) resulted in fine-grained structure in the nugget zone (Figure 12 (b)). Conversely, as the heating rate to the solution heat treatment temperature decreased, the grain structure becomes coarser and the grain growth behavior more discontinuous. With the slower heating rate in an air furnace, the grain size grew to more than 1 mm in many locations after anneal/SHT 1 and anneal/SHT 3 as shown in Figure 12 (a) and Figure 12 (c).



(a) **(b)** **(c)**

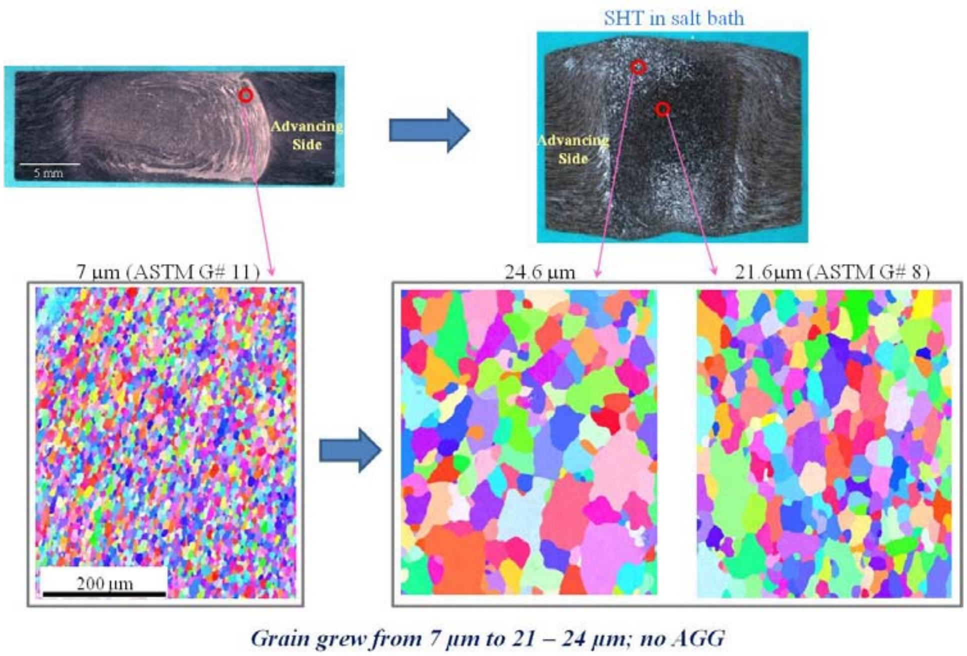
Figure 12. Effect of heating rate to the SHT temperature on the macrostructure of weld 1 specimens compressed to 15%: (a) anneal/SHT 1; (b) anneal/SHT 2; and (c) anneal/SHT 3.

The microstructures of the 39 % strained specimens are presented in Figure 13. Rapid heating in a salt bath resulted in the retention of the fine-grained structure in the nugget zone (Figure 13 (b)). The grain size grew from 7 µm to only 21 – 24 µm after SHT in a salt bath as shown in the EBSD maps (Figure 14 b). Conversely, as the heat rating to the solution heat treatment temperature decreased, the grain structure becomes progressively coarser and the grain growth behavior more discontinuous. With the slower heating rate air furnace, the grain size grew from 7 µm to more than 500 µm after anneal/SHT 1 and anneal/SHT 3 as shown in Figure 13 (a) and Figure 13 (c).



(a) (b) (c)

Figure 13. Effect of heating rate to the SHT temperature on the macrostructure of weld 1 specimens compressed to 39% strain: (a) anneal/SHT 1; (b) anneal/SHT 2; and (c) anneal/SHT 3.



(a) (b)

Figure 2. Grain size of the nugget zone in weld 1: (a) prior to anneal/SHT with 0% strain; and (b) after anneal/SHT 2 with 39% compression strain.

It is significant to note that there was no significant differences in the weld microstructure between anneal/SHT 1 and anneal/SHT 3. Both anneal/solution heat treat schedules used a conventional air furnace for the anneal portion of the schedule (R/C heated from 600°F to 800°F, annealed for 4 h). SHT 1 was then R/C heated from 800°F to 950°F and solution heat treated in the air furnace while SHT 3 used a salt bath for rapid heating from 800° to 950°F and solution heat treatment. One explanation is that the slow heating rate to 800°F and holding for 4 hours at 800°F during the air furnace anneal may have activated the AGG. Consequently, rapid heating in a salt bath to the solution heat treatment was unable to retain the fine-grained nugget structure because AGG was already present prior to the salt bath heat treatment.

In the nugget of 2195 FSW, there is a high density of soluble particles that will become unstable and dissolve at temperatures between 850 – 950°F (see Figure 11). When the heating rate is slow, the heterogeneous dissolution of unstable particles can activate AGG more progressively, allowing a few grains to grow preferentially. Thus AGG is activated during the slow heat up to the solution heat treatment. Conversely, rapid heating would facilitate a more uniform dissolution of the second phase particles and the growth of many grains can initiate simultaneously, resulting in a much finer and more uniform grain structure.

7. Effect of prior deformation strain and heating rate to the SHT temperature (using a conventional air furnace)

Although salt bath SHT shows promise in mitigating AGG within the weld nugget, it is not practical for heat treating a large rocket component such as a 18 foot diameter dome. Therefore, additional fast ramp rate SHT was conducted using a conventional air furnace. Samples were also deformed to a total of 15% strain and 39% strain prior to the SHT to study the influence of prior plastic strain on AGG. The test matrix for studying the effects of prior deformation strain and heating rate to the SHT temperature on weld microstructural evolution is shown in Table 8. Three different anneal/SHT schedules were used as shown in Table 9.

Table 8. Test matrix for studying the effects of prior deformation strain and heating rate to the SHT temperature on the weld microstructural evolution.

Weld	1st Strain Increment	Anneal	2nd Strain Increment	Total Strain	Solution Heat Treatment†	Macrostructure
Weld 1	15%	no	-	15%	Preheat/SHT 1	Grain growth/ AGG
Weld 1	15%	no	-	15%	Preheat/SHT 2	Grain growth/ AGG
Weld 1	15%	no	-	15%	Preheat/SHT 3	Grain growth/ AGG
Weld 1	15%	no	28%	39%	Preheat/SHT 1	Fine grain/ limited grain growth
Weld 1	15%	no	28%	39%	Preheat/SHT 2	Fine grain/ limited grain growth
Weld 1	15%	no	28%	39%	Preheat/SHT 3	Fine grain/ limited grain growth

Table 9. The heat treatment schedule used for compression tested specimens.

Solution Heat Treatment	Schedule
Preheat/SHT 1	725°F/15 min + R/C Heating to 950°F in 30 minutes + 950°F/1h + WQ
Preheat/SHT 2	725°F/15 min + R/C Heating to 950°F in 45 minutes + 950°F/1h + WQ
Preheat/SHT 3	800°F/15 min + R/C Heating to 950°F in 15 minutes + 950°F/1h + WQ

The effect of heating rate to the SHT temperature on the microstructure of the weld samples with 15% prior deformation strain are shown in Figure 15. For the samples that have 15% strain, there is a substantial increase in grain size throughout the entire nugget zone after the fast ramp rate SHT. The weld nugget exhibited AGG regardless of the heating rate to the SHT temperature.

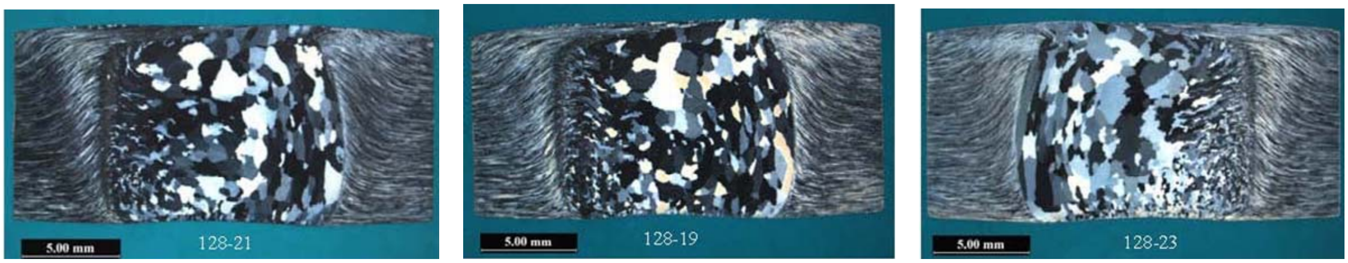
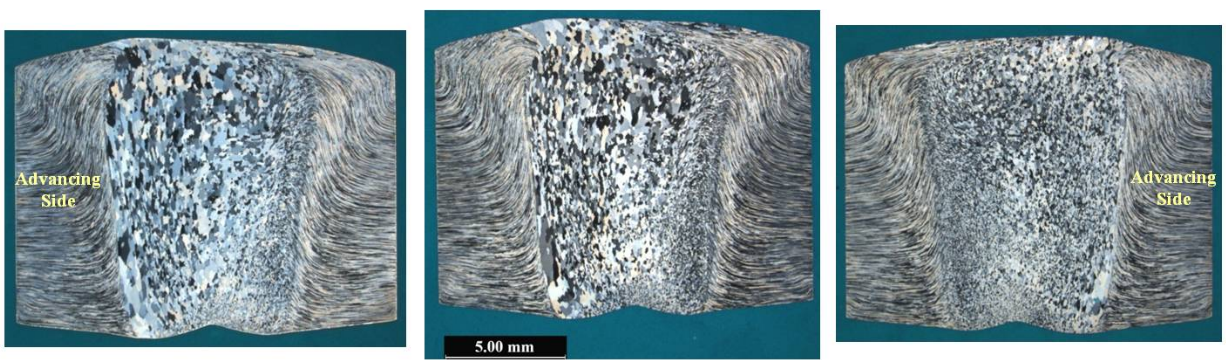


Figure 15. Effect of heating rate to the SHT temperature on GR11 weld samples with 15% prior deformation strain; (a) preheat/SHT 1; (b) preheat/SHT 2; and (c) preheat/SHT 3.

For weld samples with 39% prior deformation strain, the fast ramp rate SHT in a conventional air furnace was able to prevent AGG (Figure 16). Although there was some grain growth in the weld, the weld nugget exhibited AGG regardless of the heating rate to the SHT temperature. Preheat/SHT 3, which has the fastest ramp rates, produces the smallest grain size (Figure 16 c). So the results clearly indicate that with adequate plastic strain, preventing AGG is possible by increasing the heating rate to SHT in a convention air furnace.



(a) (b) (c)

Figure 3. Effect of heating rate to the SHT temperature on GR11 weld samples with 39% prior deformation strain: (a) preheat/SHT 1; (b) preheat/SHT 2; and (c) preheat/SHT 3.

It is apparent that that the weld grain size after fast ramp SHT in an air furnace is both prior deformation strain and heating rate dependent. A strong influence of prior strain on the microstructure was found. To avoid AGG or substantial grain growth, the welds must be deformed to a minimum strain that is greater than 15%.

The forming degree for the spun formed domes varies from 5 to 40% and therefore, the spin forming process is not conducive to producing a consistently high forming strain level. The results from this study suggest that while the fine-grained nugget zone can be retained in the high forming degree regions of the dome, it may not be possible to prevent the AGG in the lower forming degree regions.

An obvious solution to mitigate the AGG in the nugget zone of 2195 welds would be to increase the heating rate and the forming strain level. Unfortunately, for the spun-formed 2195 domes, having consistent high forming strain level is not realistic and the dome is too big to heat treat using a salt bath furnace. Thus weld grain size control will remain a challenge for the post-SHT spun formed domes.

IV. Conclusions

1. MT weld heat treated with MT anneal resulted in low forming range, low strain hardening exponent. The formability of MT weld can be enhanced by applying PW anneal 2, a new formability enhancing annealing treatment developed at Marshall Space flight Center.

2. The weld annealed using PW anneal 2 exhibits lower yield strength, higher forming range and much improved strain hardening exponent and strain rate sensitivity. PW anneal 2 is superior to both PW anneal 1 and MT anneal.

3. Salt bath SHT is very effective in reducing the grain size as it help arrest the onset of AGG and promote normal recrystallization and grain growth. However, heat treating a 18 foot dome using a salt bath is not practical. There is a need to find alternatives to the salt bath SHT.

4. The grain size after SHT is both prior strain and heating rate dependent. For samples that have been deformed to 39% strain, fine grained microstructure can be obtained by fast heating to 950 °F using a conventional air furnace. However, for samples that have a strain level below 15%, fine grained microstructure cannot be obtained by the fast heating method in a convention air furnace.

5. Current fast heating to SHT approach cannot produce uniform grain size in the spun-formed dome because the forming degree varies from 5% to 40%. Grain size can be kept finer in the high forming degree region. However, AGG mitigation would be very challenging in the lower forming degree regions.

V. **REFERENCES**

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Mitigating Abnormal Grain Growth for Friction Stir Welded Al-Li 2195 Spun formed Domes

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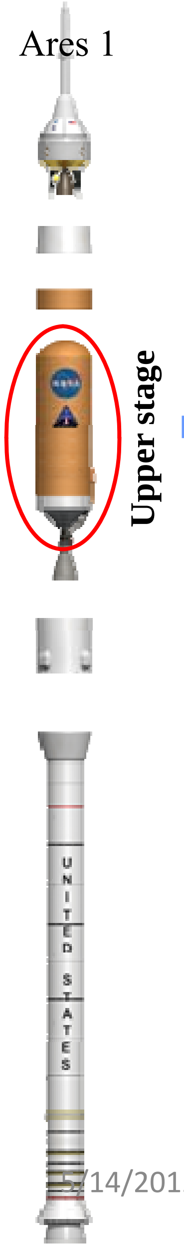
Jacobs Sverdrup ESTS Group/Qualis Corporation
NASA-Marshall Space Flight Center

May 17, 2012

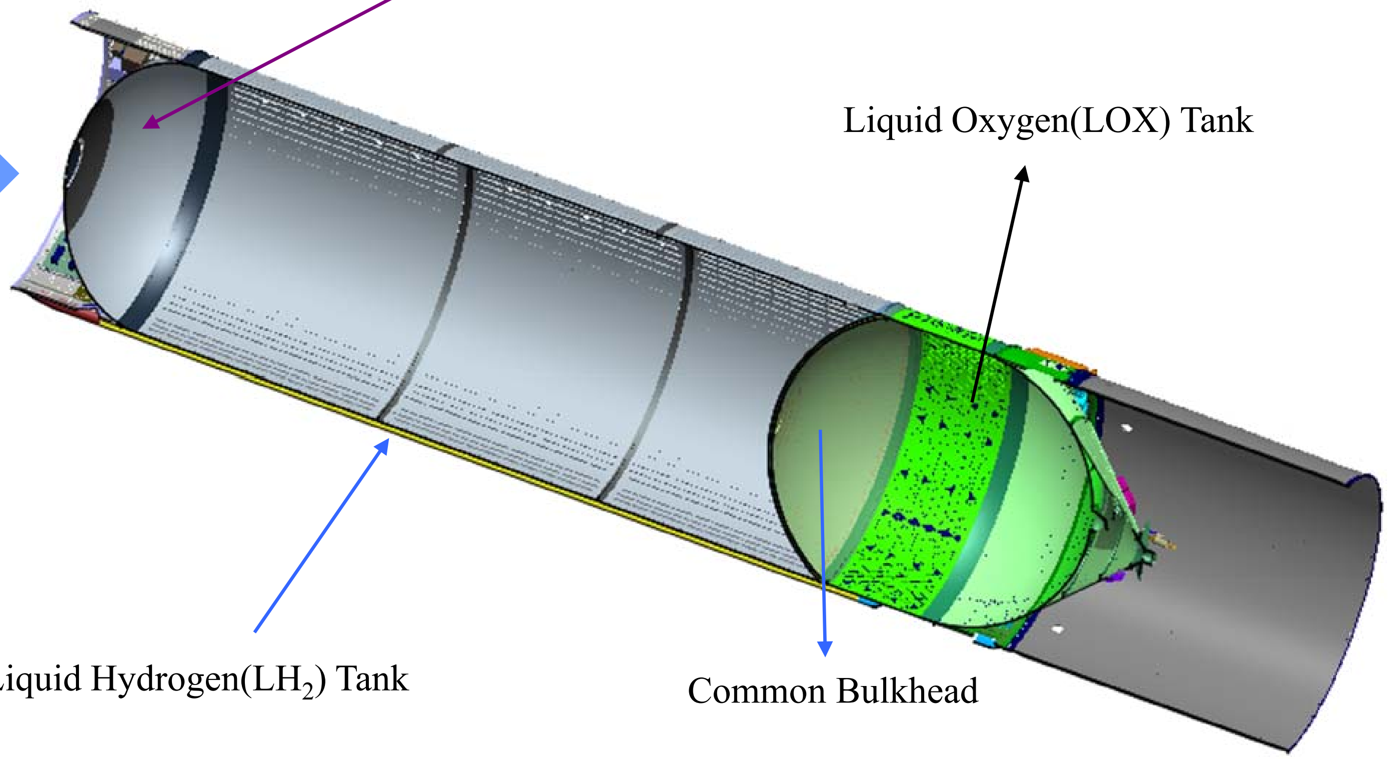
Overview

- **NASA Spun-Formed Cryogenic Dome Program**
  - Application for Ares 1 LH_2 dome
  - Gore construction vs. spin forming
- **Formability Enhancement for 2195 FSW by Increasing the Strain Hardening Exponent ("n")**
  - High strain hardening exponent can help strain distribution and increase the forming limit to prevent failure
  - Developed a novel heat treatment to enhance the formability
- **Abnormal Grain Growth (AGG) & Efforts to Mitigate AGG**
  - 2195 FSW zone is susceptible to AGG
  - AGG mechanisms & efforts to mitigate AGG
- **Accomplishments & the on-Going Development Activities at NASA-MSFC**

Dome Development for Ares 1 Upper Stage LH_2 Tank



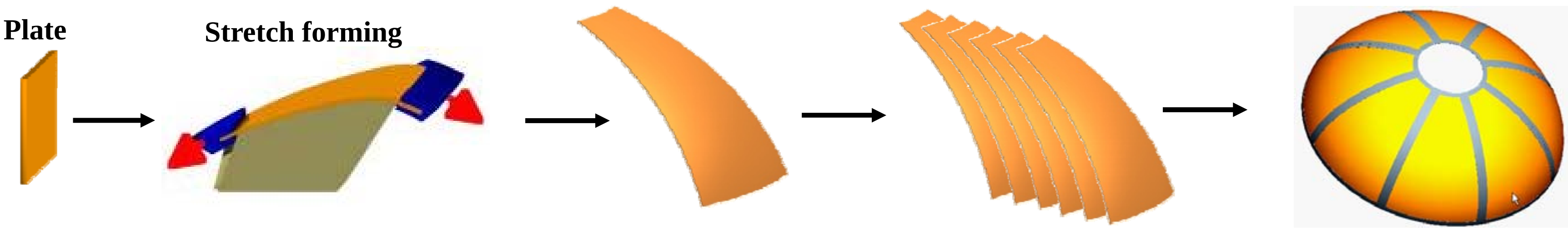
2195 dome (5 m) for LH_2 tank – can be manufactured by (1) gore construction & (2) spin forming



- 2195 dome is traditionally manufactured by gore construction, tedious & complex process
- Spun formed 2195 dome can save weight/costs; a promising new technology for the future

Gore Construction vs. Spin Forming

Traditional Approach – Gore Construction



Dome Gore Panel | Dome Gores | Welded Gore Assy. Dome

Developmental Spin forming Approach

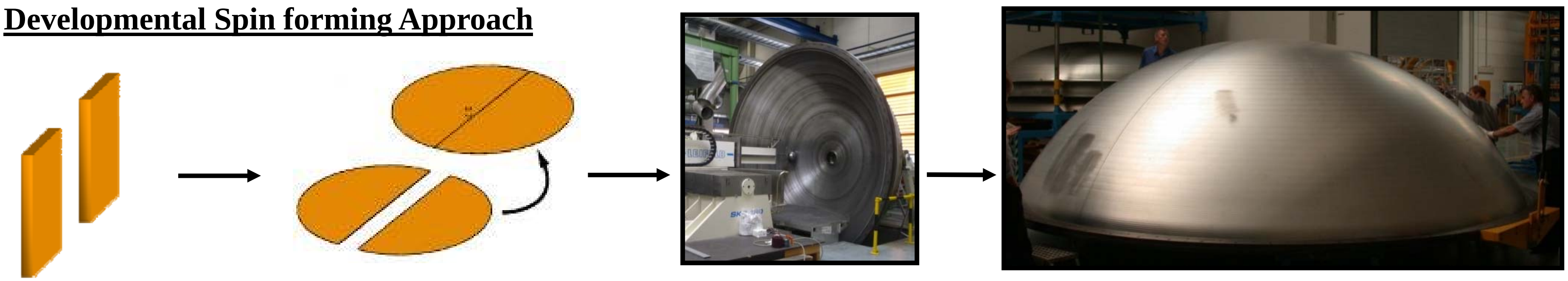


Plate | FSW dome blank | Spin forming process | Spun Formed Dome

Benefits:
- Improved safety and mission success
- Reduced part count
- Reduced weld count

Mass Savings:
- Elimination of weld lands
- Higher strength material

Cost Savings:
- Reduced material quantity
- Reduced production labor

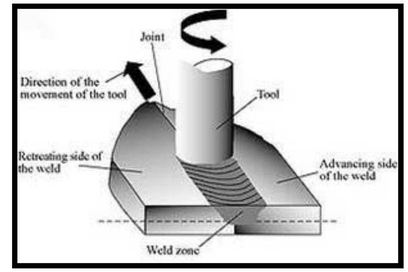
Status:
- Production application
  - Any Aluminum alloy domes
  - Up to 5.5 meter diameter

Technology Merits

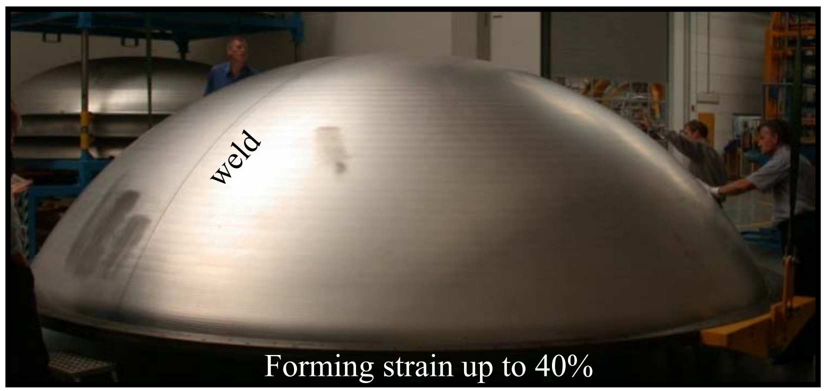
Developmental Spin forming Approach

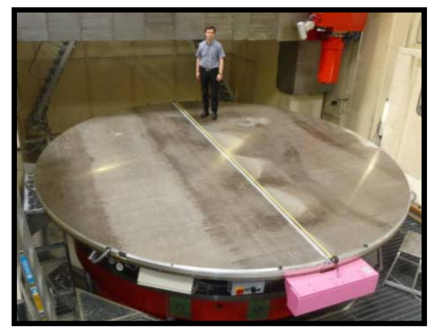
Aluminum-Lithium Alloy Plates

Friction Stir Welding
- Greater strength & ductility than fusion welding

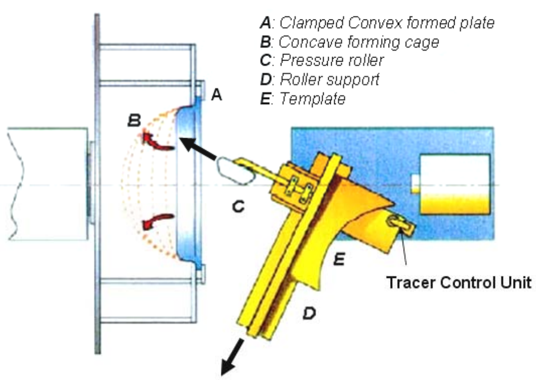


FSW Spun Formed Dome
5 m dia. Al-Li 2195





Join 2 plates into a big blank



Spin Forming
- Single piece near net shape manufacturing
- Lower manufacturing costs
- Improved mass fraction
- Improved system reliability

Two Major Challenges for the Spun-Formed Dome Development

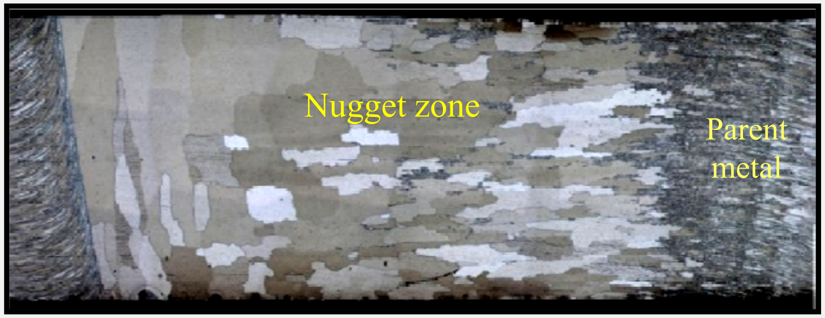
Inadequate formability

1. **Micro-cracking in the FSW**
   - Microcracks are present in the FSW
   - Microcracks make the dome susceptible to catastrophic failure
   - Developing a **formability enhancing heat treatment** to prevent microcracking in FSW

Microcracks in 2195 FSW

Weld grain size control

2. **Abnormal grain growth (AGG) in the FSW**
   - Grain size can grow from 10 µm to more than 1 mm (more than 100 x increase) in the nugget zone
   - AGG reduces the ductility/toughness to very low level
   - Working on optimizing FSW and thermo-mechanical processing parameters for AGG mitigation



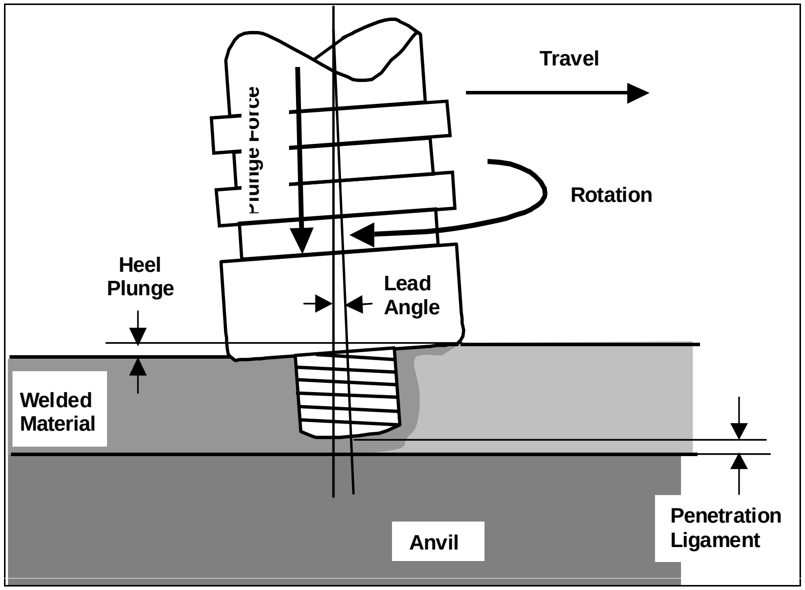
AGG in 2195 FSW

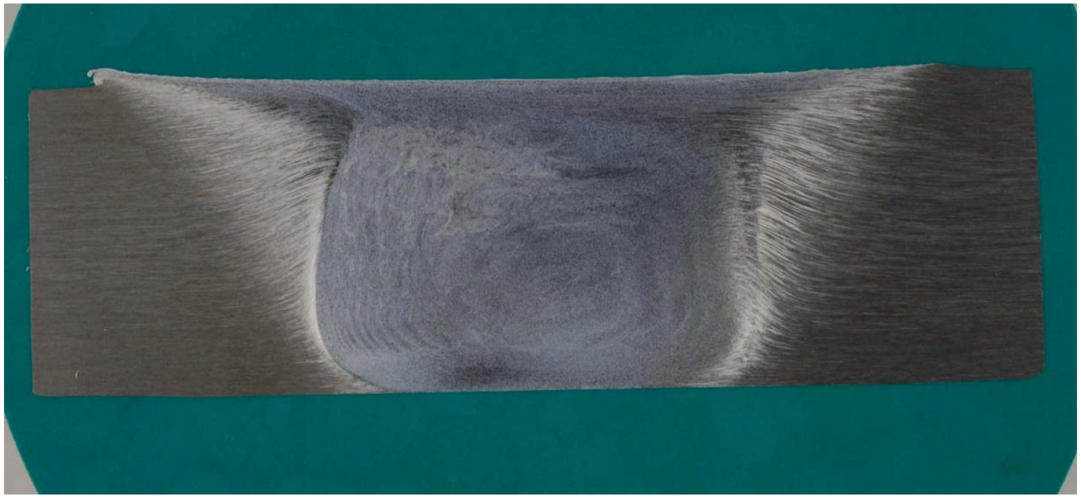
MSFC WELDED PANELS

• Panels were welded using Conventional Friction Stir Welding

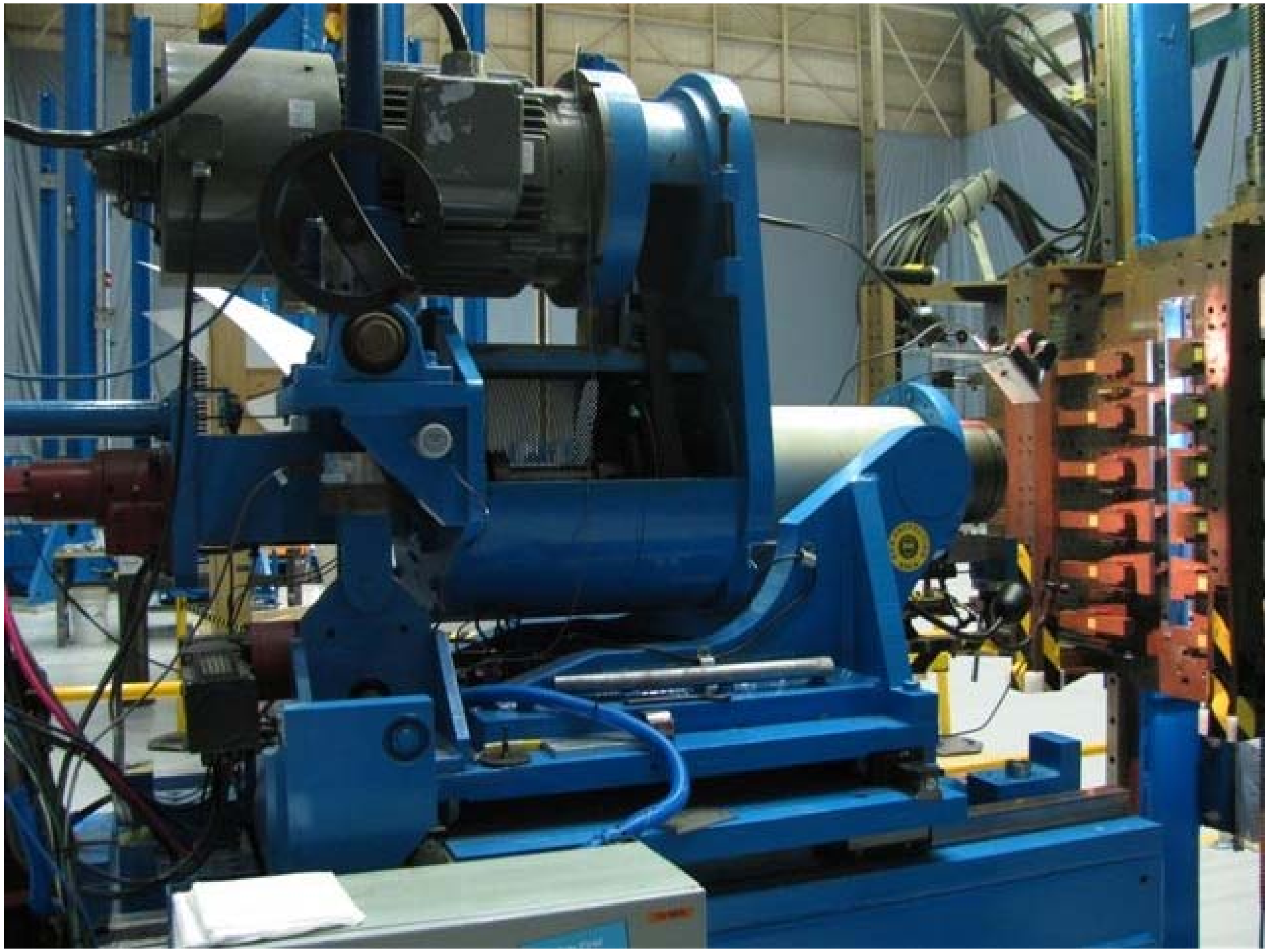
• Starting plate thickness 0.75” (19 mm) machined to 0.4” (10 mm) for welding

• Weld parameters chosen to achieve a specific nugget grain size
  - ➢ Not optimized for strength, nugget shape or appearance
  - ➢ The grain size is approximately 7.0 µm





MSFC WELDED PANELS



Horizontal Weld Tool used for welding development panels

Test Matrix for Formability Study

The tensile test matrix for weld 1 and MT weld

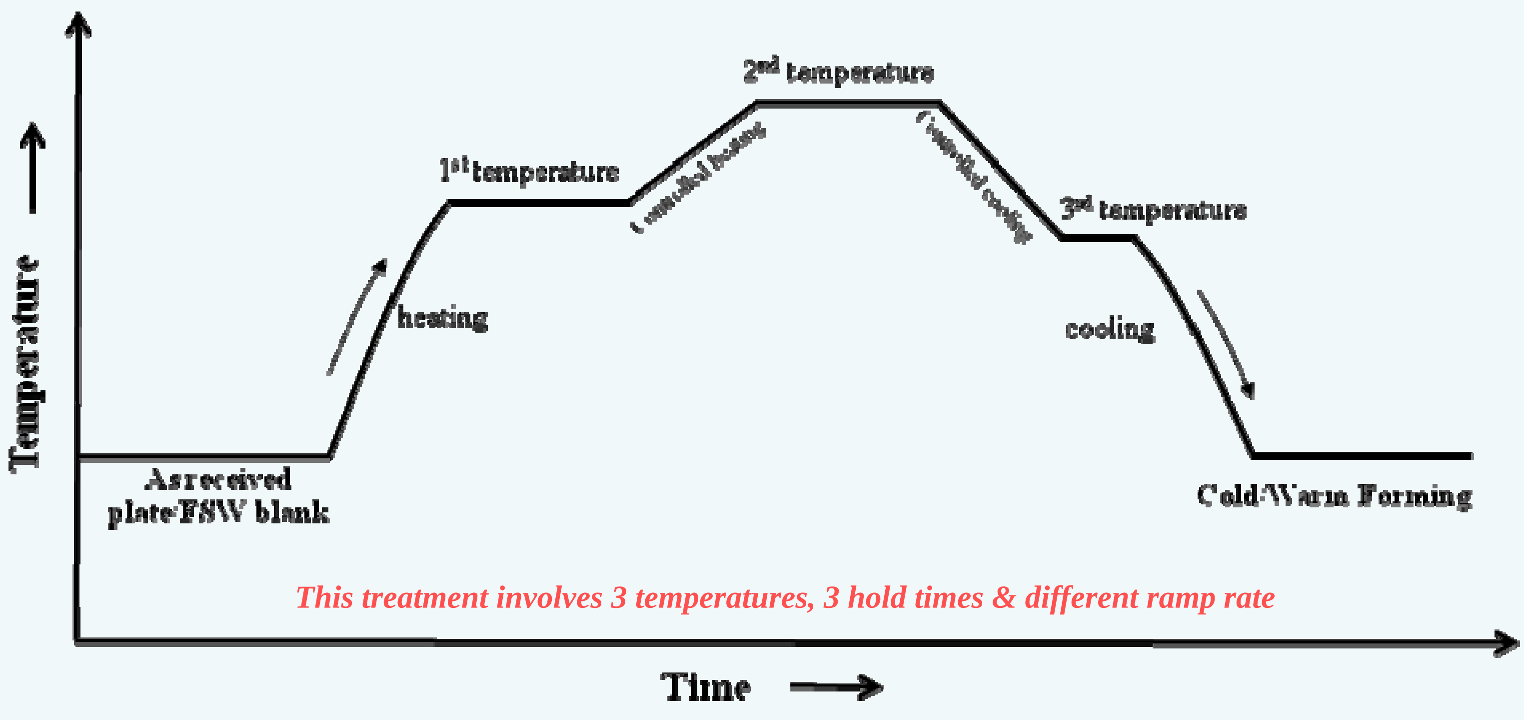
Material – **Weld 1**			
Post Weld Anneal	Tensile test Temperature	Specimen location	Parent metal Orientation
PW Anneal 1	200 °C (396 °F)	weld	LT
PW Anneal 2	200 °C (396 °F)	weld	LT

Material - **MT Weld**			
Post Weld Anneal	Tensile test Temperature	Specimen location	Orientation
MT Anneal	200 °C (396 °F)	weld	LT
PW Anneal 2	200 °C (396 °F)	weld	LT

1. PW Anneal 1: 750 F/3h + air cool
2. PW Anneal 2: A formability enhancing novel heat treatment developed by NASA
3. MT anneal: Parameters not known, panels provided by MT in annealed condition

The Formability Enhancing Novel Heat Treatment Developed at MSFC

PW anneal 2 - A multi-step ramp rate controlled heat treating process



- *Temperature, hold time, and ramp rate affect the microstructure & mechanical properties*
- *Capable of improving the strain hardening exponent for higher formability*

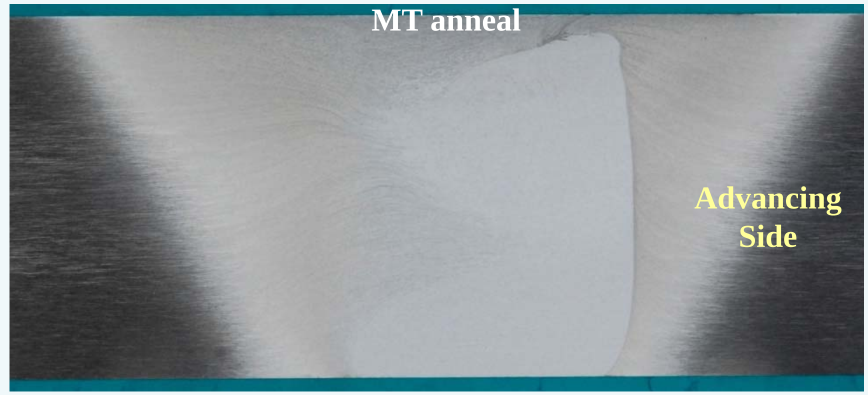
Macrostructure after Post-Weld Anneal

Weld 1

Grain size ≈ 7.0 µm

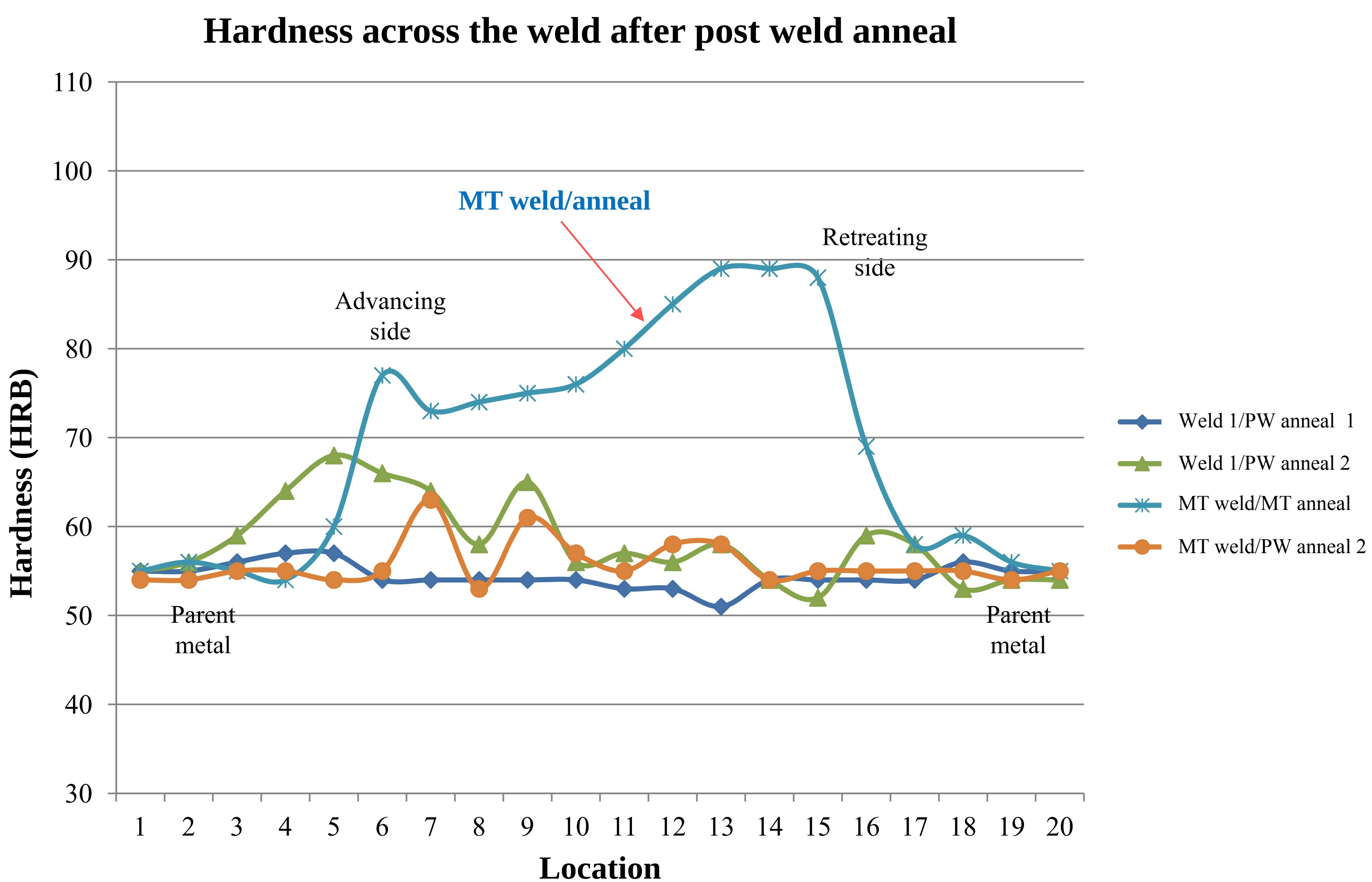
MT weld

Grain size ≈ 10.7 µm



MSFC's & MT's Weld are very different in the shape and size of the weld nugget

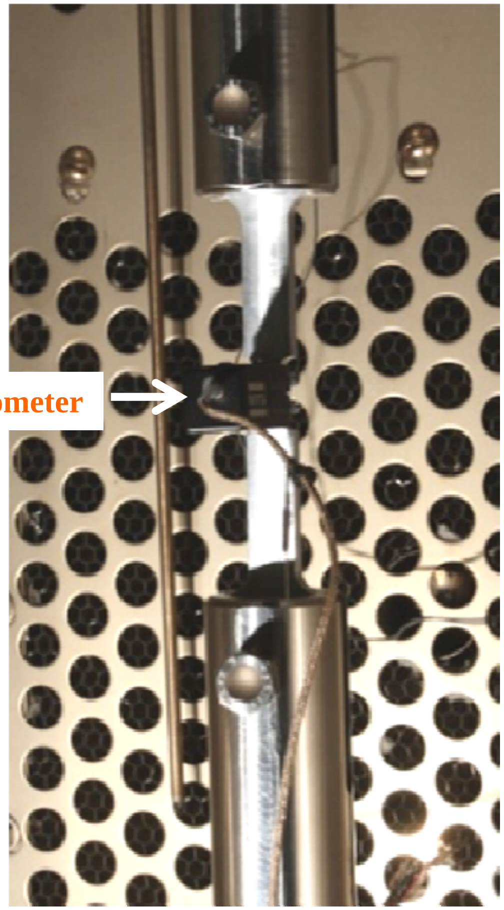
Hardness Comparison after Post Weld Anneal



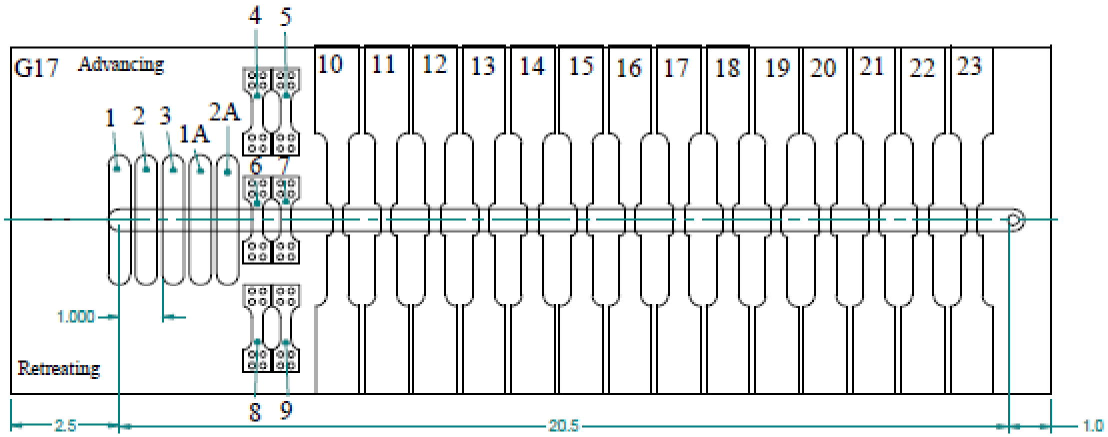
- ***MT weld/anneal has the highest hardness in the weld nugget***
- ***PW anneal 2 can equalize the weld/parent metal hardness***

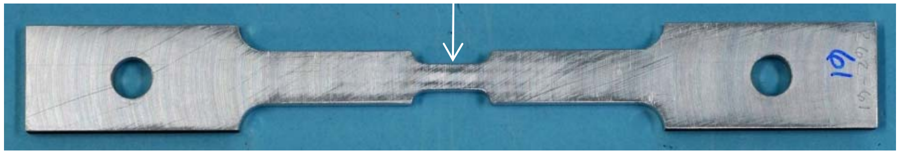
Cut Plans, Sample Configuration, and Tensile Testing

Sample with an extensometer on



Cut plan & specimen configuration





1. A double reduced gage section was used to make sure the specimen necked and broke in the nugget of the weld.
2. 0.3" extensometer was used to record strain within the 0.5" gage section of the specimen.

Effects of Post Weld Anneal on Tensile Properties/Strain Hardening

The tensile test results

Material	Anneal	Yield Stress (KSI)	Tensile Stress (KSI)	Fracture Elongation (%)	*Forming Range	Formability Ranking
Weld 1	PW anneal 1	11.5	16.6	65.9	***5.1***	*3*
Weld 1	PW anneal 2	9.3	18.0	63.3	***8.7***	*1*
MT Weld	MT anneal	17.7	22.0	58.2	***4.2***	*4*
MT Weld	PW anneal 2	11.5	20.0	60.8	***8.6***	*2*

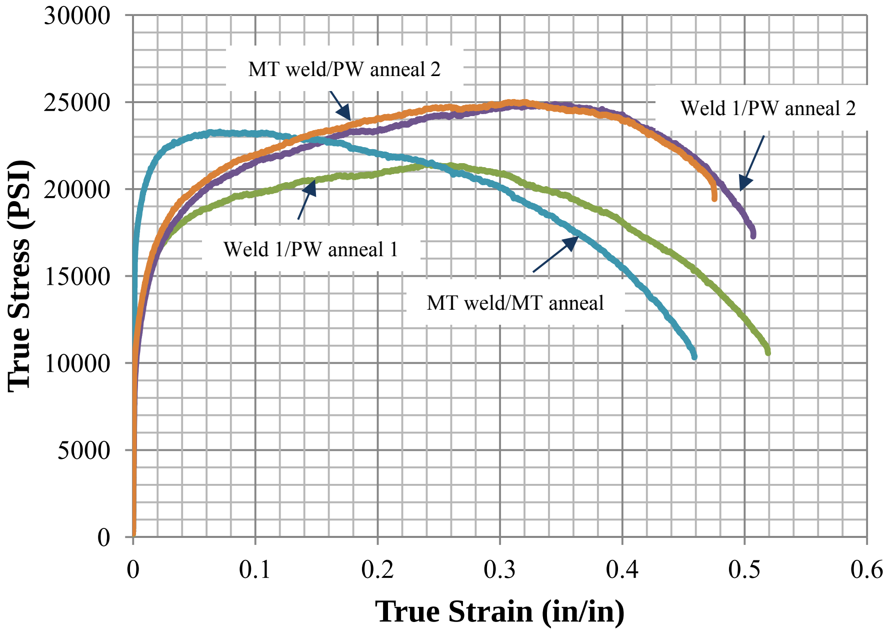
* Forming range = UTS -YS

Material	Anneal	**Strain Hardening, n (1 to 5% strain)**	**Strain Hardening, n (5 to 9% strain)**	Formability Ranking
Weld 1	PW anneal 1	0.16	0.1	*3*
Weld 1	PW anneal 2	0.21	0.15	*1*
MT Weld	MT anneal	0.08	0.01	*4*
MT Weld	PW anneal 2	0.19	0.15	*2*

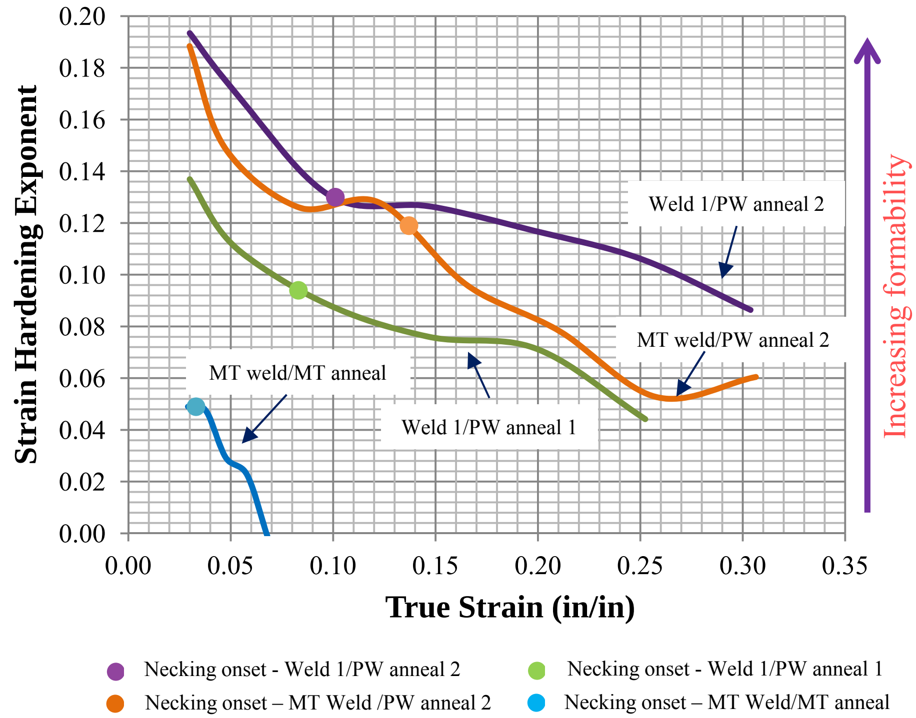
1. Formability can be enhanced by using PW anneal 2
2. PW anneal 2 produces the highest formability – highest forming range & “**n**”
3. MT anneal leads to the highest yield strength but the lowest formability

True Stress-Strain Curve Comparison (0.05 in/min)

True Stress – True Strain Curve (0.05 in/in/min)



Strain Hardening Exponent Analysis (0.05 in/in/min)



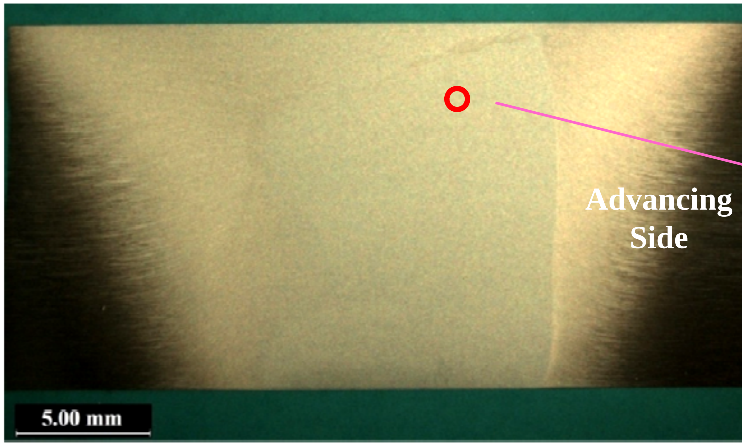
- The annealing parameters have profound effects on the strain hardening behavior
- MT anneal leads to high yield strength, early necking, and accelerated strain softening
- PW anneal 2 has the most strain hardening effect; followed by PW anneal 1 and MT anneal

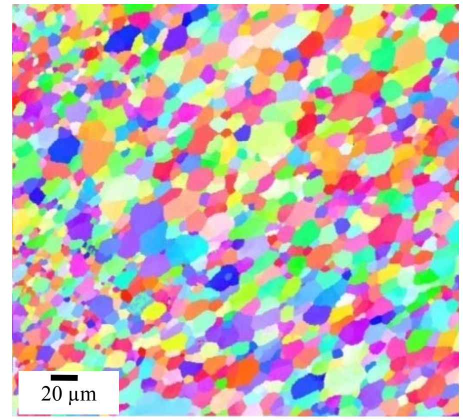
Summary of Formability Study

- **MT weld heat treated with MT anneal resulted in low forming range, low strain hardening exponent**.
  - The formability of MT weld can be enhanced by applying PW anneal 2, a formability enhancing annealing treatment developed at Marshall Space Flight Center.
- **Demonstrated feasibility to increase formability by using a newly developed anneal**
  - The “n” can be increased by more than 50%
  - Capable of promoting more homogeneous forming strain distribution
- **The weld annealed using PW anneal 2 exhibits higher forming range and much improved strain hardening exponent**
  - The current post weld anneal (MT anneal) resulted in very low strain hardening exponent
  - Low strain hardening exponent decreases the forming limit

Abnormal Grain Growth in Spun-Formed 2195 FSW

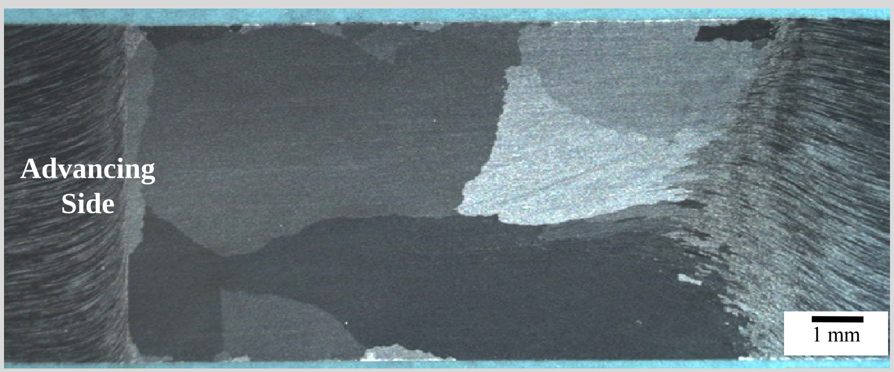
Post weld annealed – prior to spin forming

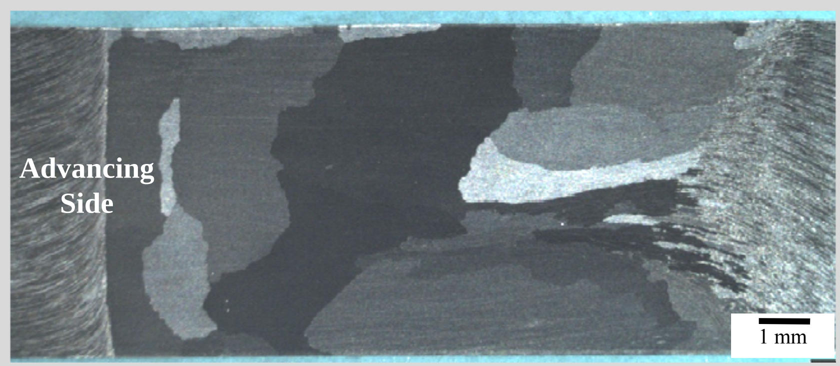




Average grain size : 10.6 µm

Spun-formed & Solution heat treated





- Grains can grow from 10 µm up to several mm in the weld after SHT
- Low ductility is a big concern; Ductility in the AGG weld generally less than 1%

AGG Drastically Reduces Ductility/Fracture Toughness

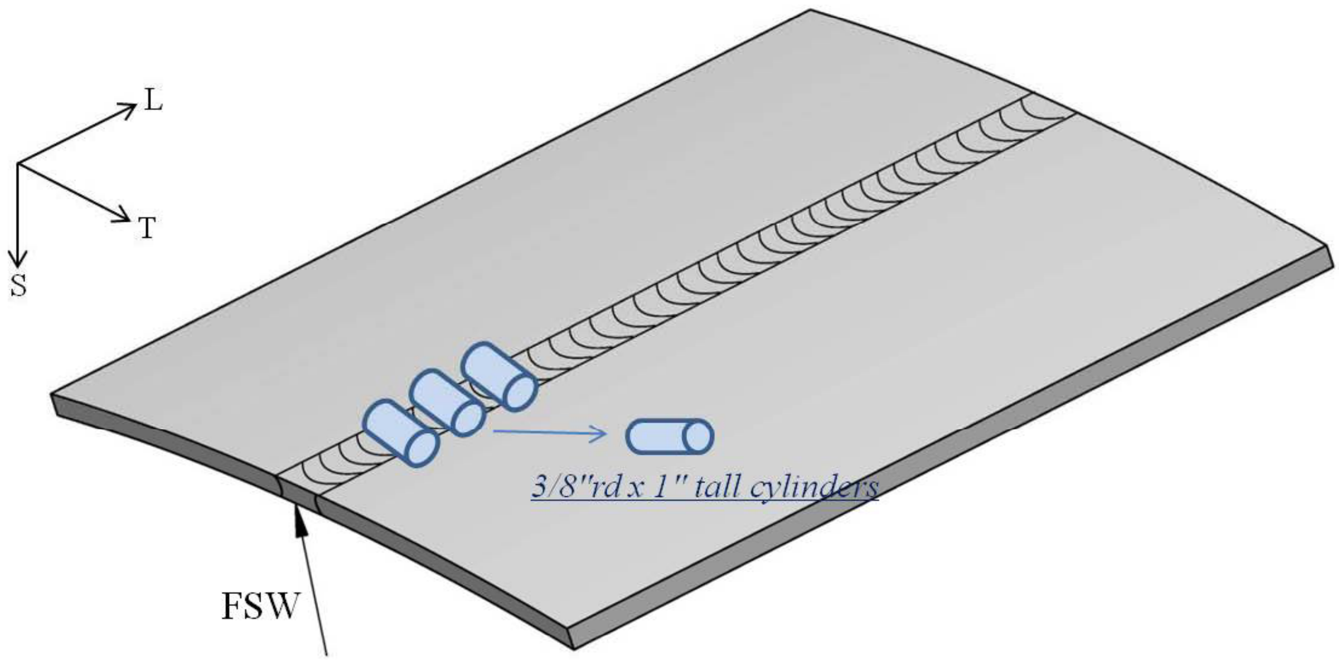
Considerable ductility reduction when AGG is present in the FSW

	Average Base metal, 45 deg orientation			Average In FSW (across weld)			
Dome/goals	UTS (ksi)	YS (ksi)	e(tot) (%)	UTS (ksi)	YS (ksi)	e(tot) (%)	
SubScale Dome 1	71.88	65.28	**9.18**	69.29	66.65	**1.11**	AGG
SubScale Dome 3	72.44	65.64	**5.23**	71.05	58.36	**5.21**	No AGG
FullScale Dome 2	75.26	65.78	13.50	N/A	N/A	N/A	

- Dome is designed based on the minimum mechanical properties
  - 1% ductility is not adequate

Material Used for the AGG Mitigation Study

Schematic of FSW blank showing orientation of the compression specimens



15% compression

30% compression

• Compression cylinder comprises both parent metal and weld
• Can see microstructure evolution at both advancing & retreating sides

Compression Straining Matrix and Heat Treatment

Compression deformation and heat treatment test matrix

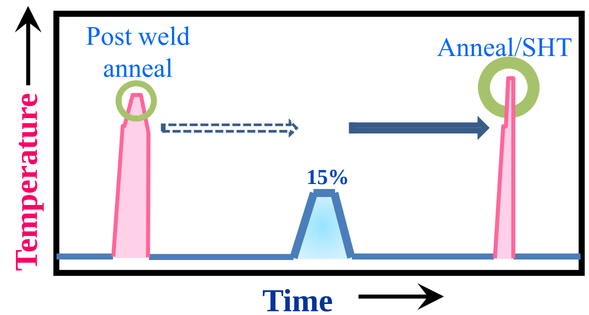
Weld	1st Strain Increment	Anneal	2nd Strain Increment	Anneal	3rd Strain Increment	Total Strain	Heat Treatment
Weld 1	15%	no	-	no	-	15%	Anneal/SHT 1
Weld 1	15%	no	-	no	-	15%	Anneal/SHT 2
Weld 1	15%	no	-	no	-	15%	Anneal/SHT 3
Weld 1	15%	no	15%	no	15%	39%	Anneal/SHT 1
Weld 1	15%	no	15%	no	15%	39%	Anneal/SHT 2
Weld 1	15%	no	15%	no	15%	39%	Anneal/SHT 3

The heat treatment schedule used for compression strained specimens

Heat Treatment	Schedule
Anneal/SHT 1	R/C heating to 800°F + 800°F/4h + R/C heating to 950°F + 950°F/1h + WQ
Anneal/SHT 2	Immersion in a 950°F salt bath + 950°F/1h + WQ
Anneal/SHT 3	R/C heating to 800°F + 800°F/4h + immersion in a 950°F salt bath + 950°F/1h + WQ

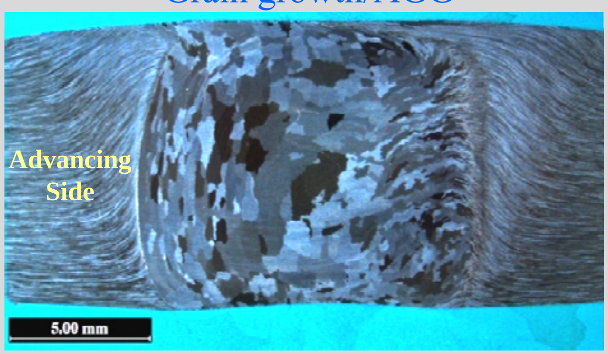
Macrostructure for 15% Strain + Anneal/SHT

Post weld Annealed - No AGG



Anneal/SHT 1

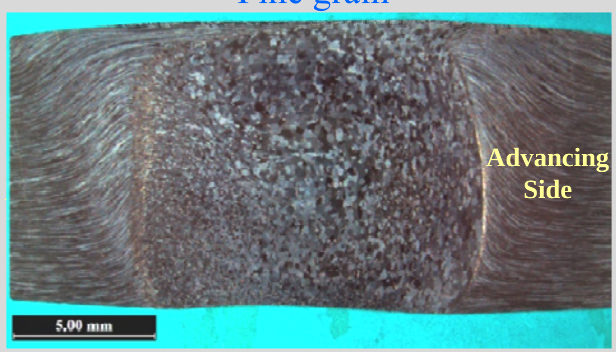
Grain growth/AGG



800 °F/4h + 15 min to 950 °F/1h

Anneal/SHT 2

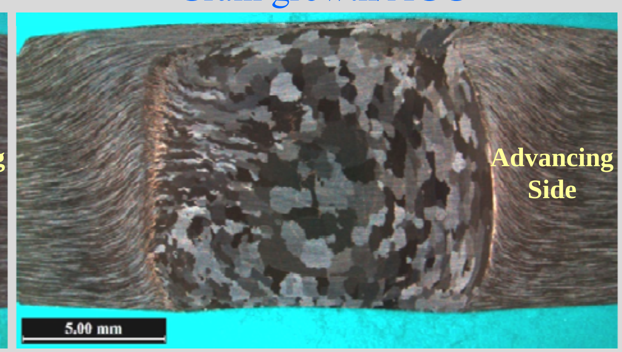
Fine grain



950 °F Salt Bath/1h

Anneal/SHT 3

Grain growth/AGG

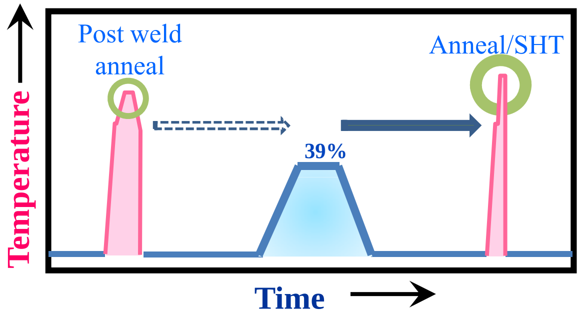


800 °F/4h + 950 °F Salt Bath/1h

Salt bath SHT can suppress AGG; Rx & gain growth starts at 800 °F

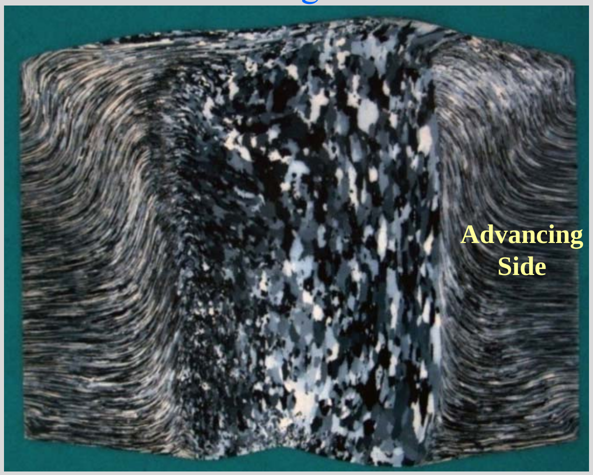
Macrostructure for 39% Strain + Anneal/SHT

Post weld Annealed - No AGG



Anneal/SHT 1

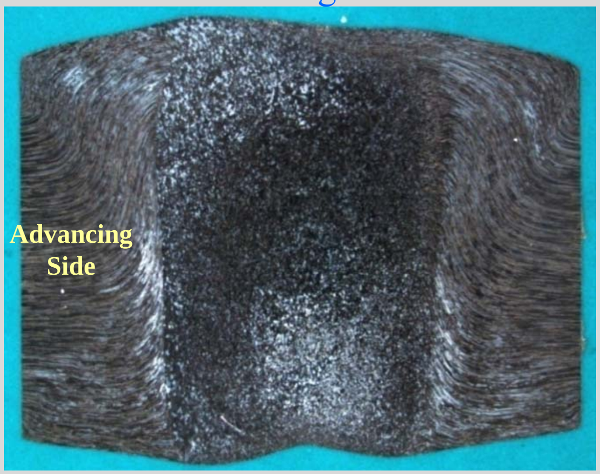
Grain growth



800 °F/4h + 15 min to 950 °F/1h

Anneal/SHT 2

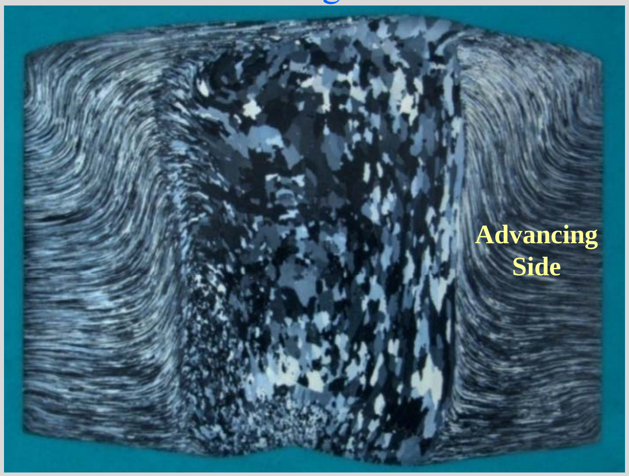
Fine grain



950 °F Salt Bath/1h

Anneal/SHT 3

Grain growth



800 °F/4h + 950 °F Salt Bath/1h

Salt bath SHT can suppress AGG; Rx & gain growth starts at 800 °F

Effects of Heating Rate and prior deformation on Microstructure Evolution

Compression deformation and heat treatment test matrix

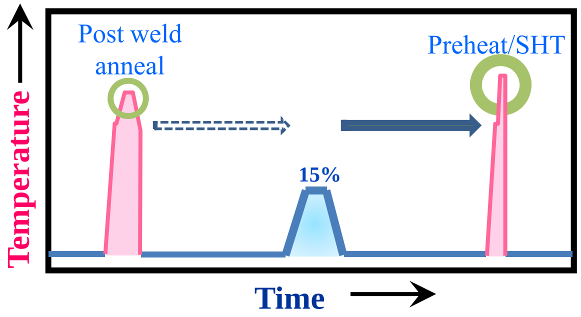
Weld	1st Strain Increment	Anneal	2nd Strain Increment	Total Strain	Solution Heat Treatment†	Macrostructure
Weld 1	15%	no	-	15%	Preheat/SHT 1	Grain growth/ AGG
Weld 1	15%	no	-	15%	Preheat/SHT 2	Grain growth/ AGG
Weld 1	15%	no	-	15%	Preheat/SHT 3	Grain growth/ AGG
Weld 1	15%	no	28%	39%	Preheat/SHT 1	Fine grain/ limited grain growth
Weld 1	15%	no	28%	39%	Preheat/SHT 2	Fine grain/ limited grain growth
Weld 1	15%	no	28%	39%	Preheat/SHT 3	Fine grain/ limited grain growth

The heat treatment schedule used for compression strained specimens

Solution Heat Treatment	Schedule
Preheat/SHT 1	725°F/15 min + R/C Heating to 950°F in 30 minutes + 950°F/1h + WQ
Preheat/SHT 2	725°F/15 min + R/C Heating to 950°F in 45 minutes + 950°F/1h + WQ
Preheat/SHT 3	800°F/15 min + R/C Heating to 950°F in 15 minutes + 950°F/1h + WQ

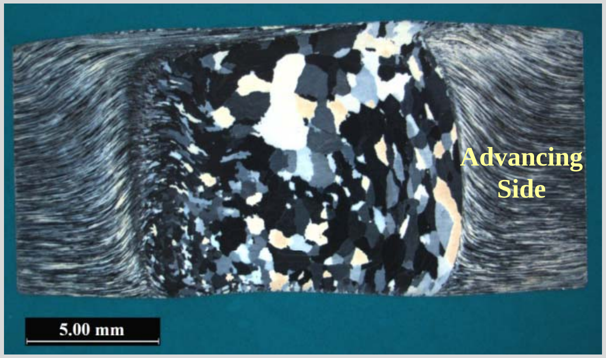
Macrostructure for 15% Strain + Preheat/SHT (FSW-1)

Post weld Annealed - No AGG



After Preheat/SHT

Grain growth/AGG

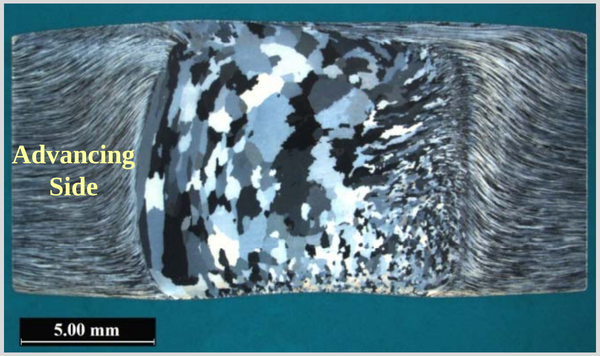


725 °F/15 min + 30 min to 950 °F/1h

Grain growth/AGG

725 °F/15 min + 45 min to 950 °F/1h

Grain growth/AGG

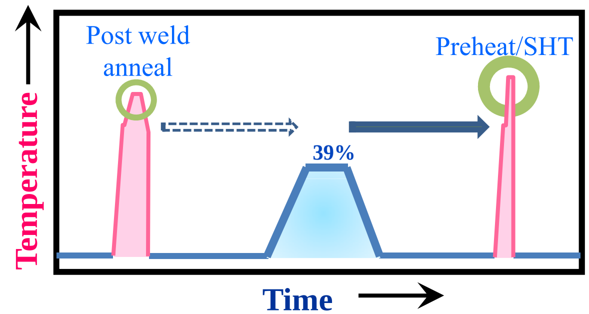


800 °F/15 min + 15 min to 950 °F/1h

Grain growth/AGG in all samples

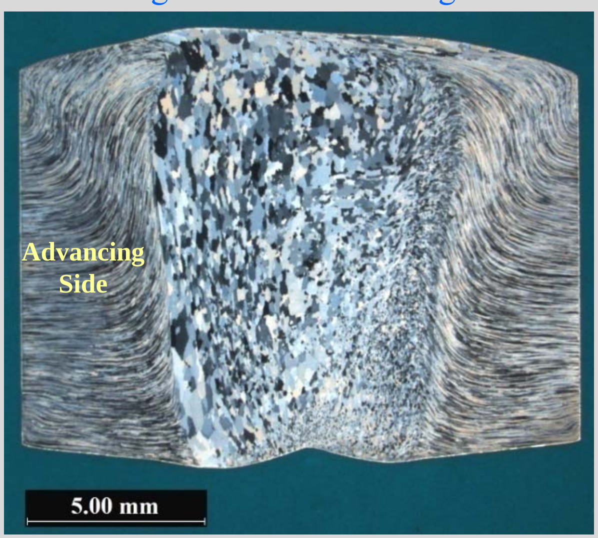
Macrostructure for 39% strain + Preheat/SHT (FSW-1)

Post weld Annealed - No AGG



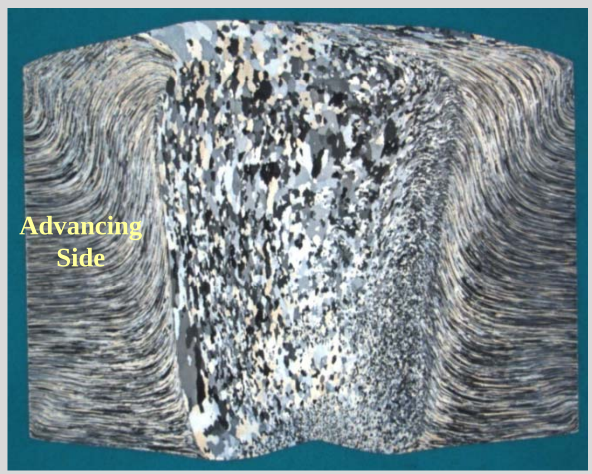
After Preheat/SHT

Fine grain/Limited Grain growth



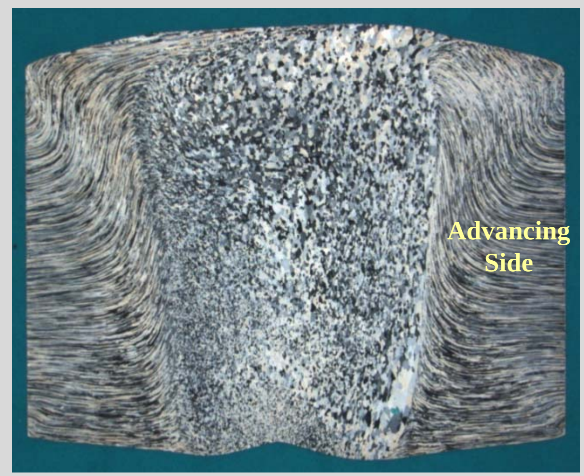
725 °F/15 min + 30 min to 950 °F/1h

Fine grain/limited Grain growth



725 °F/15 min + 45 min to 950 °F/1h

Fine grain



800 °F/15 min + 15 min to 950 °F/1h

Fine grain/limited grain growth in all samples

Summary for AGG Mitigation Study

- **Salt bath SHT is very effective in reducing the grain size**
  - However, heat treating a 18 foot diameter dome using a salt bath is not practical

- **Grain size after SHT is both prior strain and heating rate dependent. For samples that have been deformed to 39% Strain**
  - Fine grained microstructure (limited grain growth) can be obtained by fast ramp heating to 950 °F
  -

- **However, for samples that have been deformed to 15% Strain**
  - Fine grained microstructure can not be obtained by fast ramp heating to 950 °F
  - AGG is clearly forming degree (total strains) dependent

- **Current "fast ramp rate to 950 °F" approach can not produce uniform grain size in the spun-formed dome**
  - Forming degree varies from 5% to 40% in spun formed domes
  - Grain size can be kept finer in the high forming degree region
  - However, AGG probably can not be prevented in the low forming degree region (below 15%) using the current heat treating technique