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Characterization of Superplastically Formed Friction Stir Weld in Titanium 6AL-4V: Preliminary Results

D.G. Sanders, M. Ramulu, E.J. Klock-McCook, P.D. Edwards, A.P. Reynolds, and T. Trapp

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A collaborative University and Industry research project was undertaken to evaluate the performance of as friction stir welded (FSW) and friction stir welded and superplastically formed (FSW-SPF) titanium 6Al-4V alloy sheets. The purpose of this initial phase of research is to test and evaluate the mechanical properties of FSW and post SPF-FSW 2-3 mm thick sheets of titanium. As-FSW and FSW with SPF Ti alloy (standard and fine grain size) butt joints were characterized in terms of microstructures, micro-hardness, metallurgy and tensile properties. The preliminary results of the FSW and post FSW-SPF joint were found to be close to that of as-received titanium with respect to strength, but elongations were decreased.

Keywords fine grain, friction stir welding, micro-hardness, microstructure, super plastic forming, tensile properties, titanium, 6Al-4V

1. Introduction

Friction stir welded (FSW) is a cutting edge technology that permits a butt weld to be used where traditional methods for such a weld would be unacceptable. A TIG fusion weld melts the material, which can result in extremely large grain structure in the weld nugget, voids, surface defects and cracks associated with thermal contraction due to cooling. However, the friction stir weld is completely solid state. The resulting weld is much stronger and more reliable than a traditional weld. FSW can also be used on very thin sheets of material.

In the FSW process, a rotating tool consisting of a shoulder and a probe is plunged into the interface of two materials and then translated along the joining line as shown in Fig. 1. The shoulder contacts the surface of the two materials and as a result of high rotational speeds and a vertically applied forge load, a large amount of frictional heat is created. This frictional heat softens the material and extrudes it around the tool pin, mixing the two materials together (Ref 1, 2).

Since its inception in 1991, FSW has been successfully applied in the joining of aluminum (Ref 3-8), magnesium, copper and very recently, titanium alloys (Ref 9-12). Since titanium is such a useful material in the aerospace industry, the application of FSW to titanium is a process of much interest. Consequently, the driving force behind this research is to use FSW to weld multiple sheets of titanium together to form large blanks needed for large scale near net shape forming operations.

The super plastic forming (SPF) process has been used for over 30 years in the aerospace industry to fabricate complex shapes of parts. It permits the relatively inexpensive, fast, and high tolerance production of sheet metal monolithic structures. The process is accomplished by placing a die and a sheet metal blank into a hot press and forcing the blank into the shape of the die with carefully controlled backpressure as shown in Fig. 2. This process relies on the materials superplasticity, or ultra high plasticity, at elevated temperatures (Ref 13). When compared to traditional hot forming, SPF requires fewer fixtures and less specific hardware making the system versatile with short changeover times. The shapes possible are virtually limitless with only a new die needed for each new shape. To date SPF has been successfully applied to several materials including both aluminum (Ref 14, 15) but not titanium.

Currently, there is no reported research on the combination of these two processes in producing titanium structural components. By combining the two processes, the goal of near-net shape manufacturing of large titanium parts can be accomplished. FSW is one of the only solid state joining processes that does not destroy the superplastic nature of a material and it is for this reason that this combination of friction stir welding and superplastic forming is being investigated.

2. Experimental Methods and Procedures

2.1 Materials

All experiments were conducted using Ti 6Al-4V alloy. However, two types of this alloy were employed. The first was standard Ti 6Al-4V (8-10 micron grain size) and the second was fine grain Ti 6Al-4V (0.8-2 micron grain size). The stock sheet has the yield strength of 1100 MPa, ultimate strength of 1170 MPa and elongation of about 10% (MatWeb, 2006).

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D.G. Sanders, M. Ramulu, E.J. Klock-McCook, and **P.D. Edwards**, Department of Mechanical Engineering, University of Washington, Seattle, WA; **D.G. Sanders** and **P.D. Edwards**, The Boeing Company, Seattle, WA; **A.P. Reynolds**, The University of South Carolina, Columbia, SC; and **T. Trapp**, Edison Welding Institute, Columbus, OH. Contact e-mail: daniel.g.sanders@boeing.com.

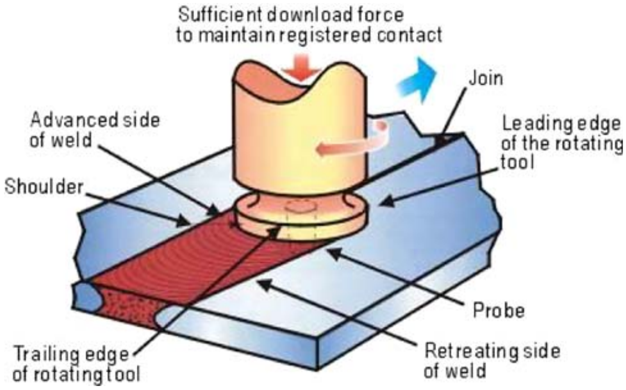

Fig. 1 Schematic of friction stir welding (Ref 2)

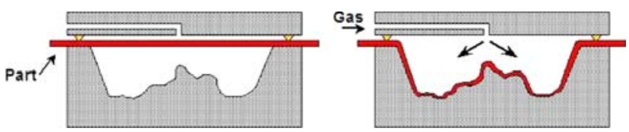

Fig. 2 Schematic of superplastic forming

2.2 Specimen Fabrication

All FSW samples were fabricated by either the Edison Welding Institute (EWI) or the University of South Carolina (USC). These samples were fabricated out of 2.03 and 2.54 mm nominal thickness Ti 6Al-4V sheet material. The sheets were roughly square and varied in size from 20.3 cm by 20.3 cm to 91.4 cm by 91.4 cm. As received FSW materials were deburred according to the best practices for the aerospace industry.

The large FSW sheets were then Superplastically formed into "Zeppelin" pans. These pans are half cylinders with closed rounded ends (the shape of half of a dirigible). However, the cylindrical walls of the zeppelin pans were not smooth like a cylinder wall as shown schematically in Fig. 3. Instead, the walls of the pans were incrementally flattened so that tensile and fatigue coupons could be fabricated from them. Furthermore, the blanks were placed into the press so that the weld line would run transversely across the center of the zeppelin pans. By using these pans, the tensile samples will have varying degrees of super plastic strain. The samples cut from the bottom area of the pan will have the most (typically 70% SPF strain) while the region at the edge will have much less (typically 40% SPF strain). This is due to the fact that the material at the bottom of the pans has to travel a farther distance, thereby deforming or elongating more than the material along the sides. Details of zeppelin pans were reported in Ref 16 and 17.

Blanks were placed into the die by hand, exposed to approximately 927 °C for 45 min at pressures from 70 kPa to 4.1 MPa and then removed by hand and allowed to cool in air. Following the actual SPF process, each pan was chemically milled as per the usual post SPF process. The purpose of this milling process is to remove the alpha-case, or oxidized outer layer of the titanium. This process removes 0.152 mm of material (Ref 18-20).

2.3 Surface and Subsurface Examination

Microstructural evaluations were done in order to examine the grain structure of the Weld Nugget (WN), the Heat Affected Zone (HAZ), and the base metal after the FSW process. To accomplish this, the samples were first cut, via abrasive water jet, from a titanium plate that had been FSW. The sample included a cross section of the weld nugget oriented perpendicularly to the joining line, shown schematically in Fig. 4. The samples were then polished on 5, 1, and finally 0.3 micron alumina-oxide polishing wheels. The sample was then etched to expose the microstructure.

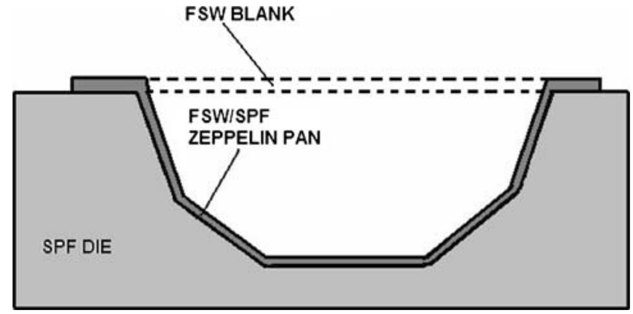

Fig. 3 Cross-sectional schematic of zeppelin pan

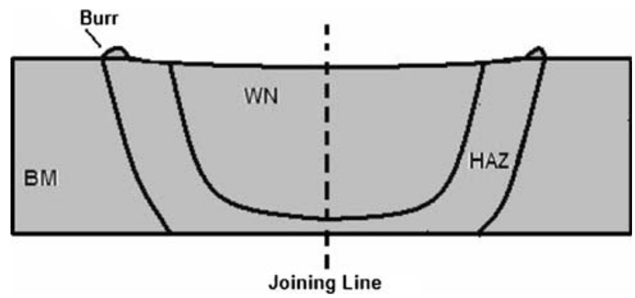

Fig. 4 Schematic of FSW cross-section showing base metal (bm), heat affected zone (haz), and weld nugget (wn)

A JEOL JSM-840A scanning electron microscope (SEM) was used in conjunction with a JEOL DSG Plus digital scan imaging system to digitally capture the SEM images. First, a series of low magnification (20×) micrographs were taken to encompass the whole sample. This was followed by a series of high magnification micrographs (300×). These were taken at approximately 1.4 mm from the bottom of the weld.

In order to determine material constituents within the weld nugget that may have been left behind by the FSW tool, a Tracor Northern TN-5402 energy dispersive X-ray analyzer (EDS) was used. Abnormal or unexpected features were then photographed using traditional SEM techniques. A 15 kV accelerating voltage was used for the EDS test which is considerably higher than the voltage for the earlier SEM tests. As a result, the SEM micrographs taken in conjunction with the EDS are not directly comparable to those taken earlier.

Micro-hardness tests were utilized to assess the surface and subsurface characteristics of both the base material and the weld nugget. These samples were prepared in the same manner as discussed for the microstructural evaluations. The samples were tested with a 100 g load applied for 15 s using a Knoop hardness tester. The first test was run at the midpoint of the cross-sectional thickness, starting in the base material and then moving to the HAZ, the weld nugget, back into the HAZ and finally into the base material again (Fig. 4). The distance between successive points was 100 microns.

The second test sought to produce a complete two-dimensional grid covering the entire cross-sectional region of interest. The sample was prepared in the same manner as above. In this test, a sample of base metal readings was taken across the thickness of the weld. Then, beginning at the edge of the HAZ, a grid of points was laid down at 100 micron intervals across the width of the weld.

2.4 Tensile Tests

The FSW plates were cut into tensile test coupons (Fig. 5) according to ASTM standard E8-04 using abrasive water jet cutter. The samples were all cut transverse to the weld so that the weld was in the center of the tensile specimens gage length. Furthermore, all specimens were fabricated parallel to the rolling (longitudinal) direction as per ASTM standards. Four to five coupons were obtained per plate. The approximate weld width and exact minimum thickness and nominal thickness of the weld were also measured. These coupons were tested on a MII 50 Unidrive load frame. A gage length of 5.08 cm for the Satec extensometer was used. Through a data acquisition system, plots of engineering stress vs. engineering strain are produced in addition to calculating tensile strength, yield strength, Young's modulus, and elongation.

3. Results

3.1 Microstructural Evaluation

In evaluating the microstructures using the SEM, two points became clear. The grain size decreased noticeably in the weld nugget when compared to the base material and there were also some defects present. The difference in grain size can be seen below. Consider the left portion of Fig. 6, which is from the base material and right which is from the center of the weld nugget.

These micrographs were taken from a standard sample of Ti 6Al-4V which has been FSW. The grain refinement is clear and is the reason that the samples thinned, or superplastically strained, at a greater rate in the weld region when undergoing superplastic forming. Also, within the weld region, a number of defects could be seen. A typical defect is shown in Fig. 7.

These defects are approximately 20-25 microns in size and were initially thought to be embedded tungsten from the FSW tool. In order to examine this further, they were scanned using the EDS system as described previously. However, none of the EDS scans showed any tungsten present. A typical EDS scan is shown in Fig. 8 and all of the elements found were expected in the titanium sample. Therefore, these defects are speculated to

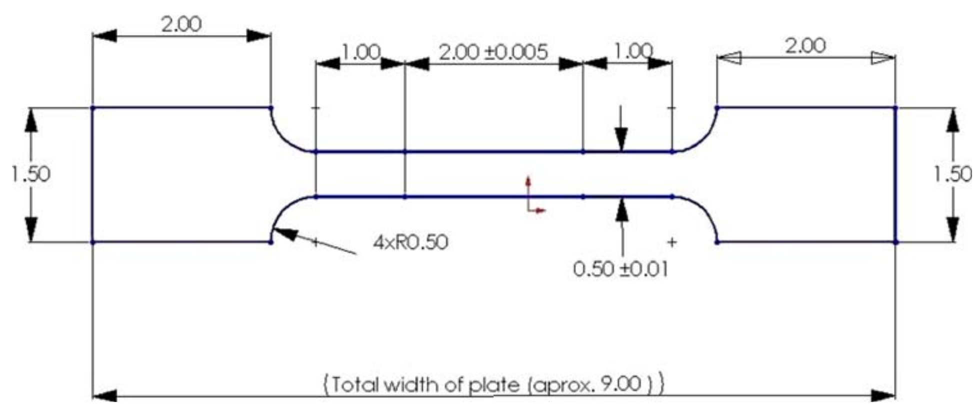

Fig. 5 Tensile specimen specifications (units are in inches)

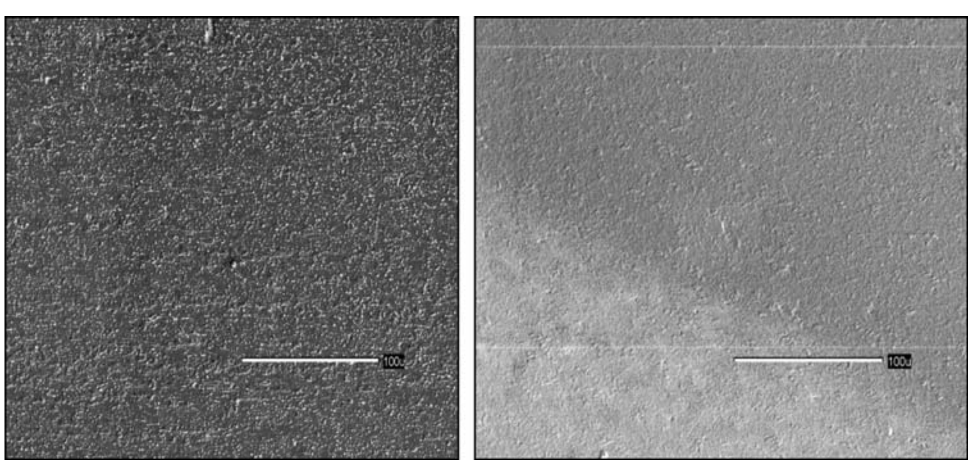

Fig. 6 Micrograph, Ti 6Al-4V, 300× base metal (left) Ti 6Al-4V, 300×, weld nugget (right)

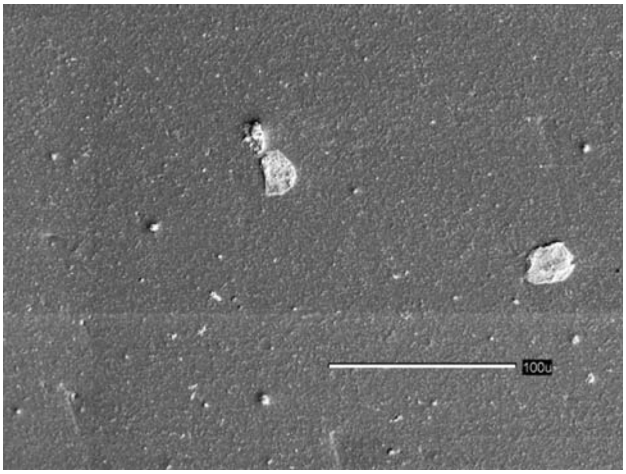

Fig. 7 SEM photograph of microstructure with typical defects

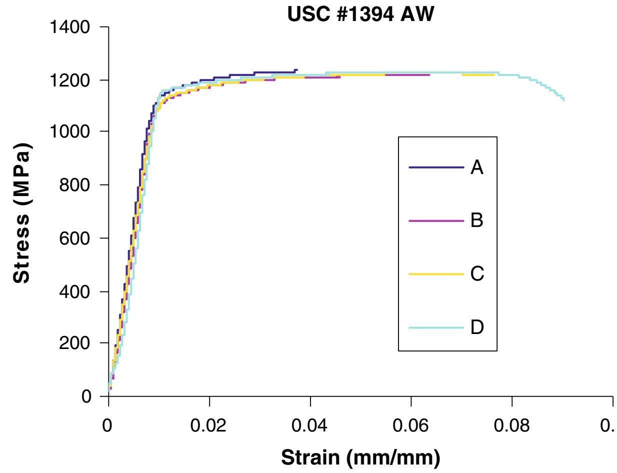

Fig. 9 Typical as FSW stress-strain curve

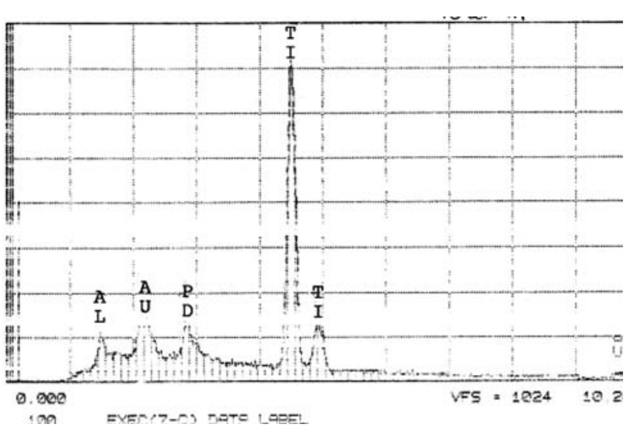

Fig. 8 Typical EDS scan showing material constituents within weld nugget

be microstructural defects rather than inclusions of tool material.

3.2 Tensile Properties

Typical stress-strain curves of FSW and FSW-SPF specimens are shown in Fig. 9 and 10, respectively. Standard as-received Ti 6Al-4V has yield strength of 1100 MPa and an ultimate tensile strength of 1170 MPa. For as FSW samples, yield strengths ranged from 817 to 1182 MPa while the ultimate tensile strengths ranged from 921 to 1224 MPa. This represents, at worst, only a 26% reduction of yield strength with the average reduction being only 8%. For the ultimate tensile strengths, the worse case reduction in properties was 21% while the average reduction was 9%. Of the past work surveyed on aluminum alloys, these results represent the best performance in a friction stir weld to date (Ref 4, 15). The post SPF samples showed a drop of only 2% below that of the as FSW. The standard deviation of the data for both the yield and ultimate tensile strengths was approximately 60 MPa with a sampling size of 61 FSW and 21 FSW-SPF specimens respectively.

Percent elongation is another important property in a metal and in this category the samples did not perform as well. Standard Ti 6Al-4V material has a typical elongation of 10% at failure. The average elongation of FSW samples was approximately 5% with a standard deviation of about 3%. The average elongation of post-SPF samples was 2.7% with a standard deviation of 1%. In both cases, this represents a significant reduction in ductility from the as received material. In the worse case the ductility was reduced by 92% and on average a 50% reduction in percent elongation was observed.

3.3 Micro-Hardness

The first micro-hardness test performed was conducted at a constant depth in the thickness of the sample at 100 micron intervals across the entire specimen. The results indicate little discernable change in hardness across the weld as shown in Fig. 11. Virtually all the data fit within one standard deviation from the mean. This test was performed on Ti 6Al-4V regular grain alloy.

The second test, which was creating a two-dimensional micro-hardness grid, was performed on a Ti 6Al-4V fine grain sample. The results were normalized against hardness measurements from base material at the same depth and plotted in Fig. 12. Therefore, a value of one is equal to the hardness of the base material, a value less than one is softer than the base material, and a value greater than one is harder than the base material. The results of this test show that there are clear bands of equal hardness that run at a constant depth in the weld. However, at a given distance from the joining line, there does not seem to be any consistency in the hardness across the depth of the weld.

4. Discussion

4.1 Tensile Tests

Upon evaluation of the tensile test data, it is apparent that the yield and ultimate strengths of the samples held very close to the parent material, Table 1. Even in the worse case, the strength was only degraded by 26% and on average by 9%. What is crucial to note is that the regular grain FSW titanium samples displayed yield and tensile strengths approximately 6% better than the fine grain samples. However, the elongation of both types was similar and only half of standard Ti 6Al-4V.

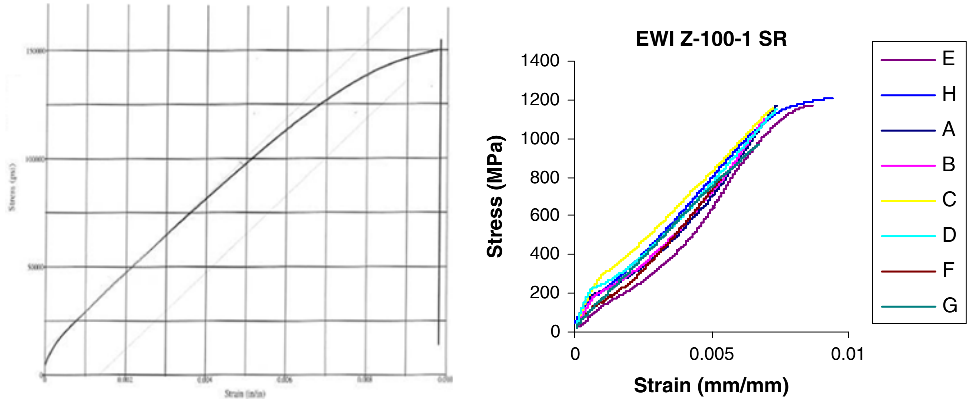

Fig. 10 Typical FSW-SPF stress-strain curve

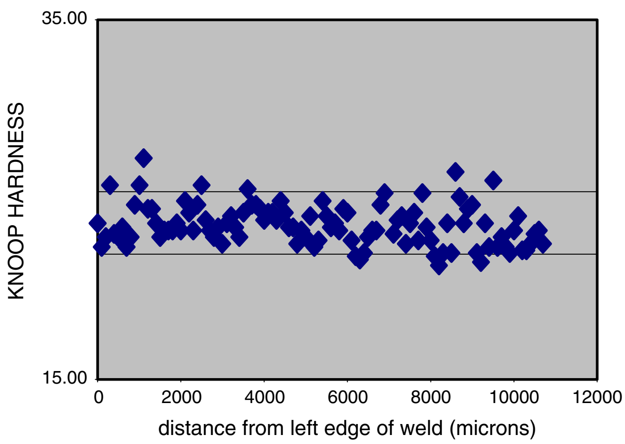

Fig. 11 Micro-hardness results (constant depth)

This reduction in ductility could be due to the disruption that the FSW causes to the grain structure in the titanium. Ductility depends on the metallic grains being able to move with respect to one another. The two factors that affect this process are grain orientation and grain size. Larger grains with common slip planes will slide more easily and smaller grains, with more randomly oriented grain boundaries, will slip less easily (Ref 20, 21).

In a standard sheet of titanium, the grains are largely aligned due to the rolling process. However, the FSW, by its very nature, stirs these grains into a random orientation. Also visible in these micrographs is the grain refinement that takes place during the FSW process. As seen in Fig. 6, due to the enormous mechanical and thermal energy placed into the sample by the welding tool, the grains are refined to a smaller size. The grain refinement and disorder is the likely cause for the observed decrease in the material's ductility. This is an important aspect of the friction stir weld because it could increase the metal's sensitivity to defects and cracks. Elongations of just 5% are bordering on brittle behavior. This means that a defect or crack could cause the piece to fail in a brittle manner which is rapid and catastrophic crack propagation.

The samples which had been super plastically formed are broken down into groups based on their degree of super plastic strain and are listed in Table 2. From this, it is apparent that the strength of the material actually increases with the amount of super plastic strain it has experienced though its ductility decreases. At a minimum, the super plastic samples perform with parity to the non-SPF samples and in some cases better. This could be due to the inherent stress relief in the SPF process.

It is known that the FSW induces residual stresses in the material. This can easily be seen when a plate that has been joined by a FSW is looked at on edge because there is a slight bow toward the top of the weld. This is the result of residual stresses. In Fig. 12, it is clear that there are bands of largely equal hardness. The hardest regions are those closest to the top of the weld. These residual stresses can result in the reduction of the material's strength. However, when the sample has been super plastically formed, those stresses are relieved. The high temperature of the press effectively anneals the material. This is the likely cause for the SPF/FSW samples being equal to or better than the FSW sample. The stress relief must at least counteract the negative effects of the FSW process.

The SPF process also has a detrimental effect. SPF samples displayed an average elongation of just over 25% that of the base metal. This is well into the brittle range and means that the material would likely be very sensitive to cracks or defects and could fail in a rapid, catastrophic manner. Part of the cause for this observation could be the necking that was present in the sample due to the SPF process. All of the samples thinned considerably in the weld region. The average weld is only 12.7 mm across and the gage length used for the test was 5.08 cm. Therefore, it is likely that the majority of the strain took place in the thinner weld region meaning that the elongation in the weld itself was significantly greater than the results indicate. This is because gage length is substantially large compared to the area of interest and thus the localized strain in the weld region may not be accurately accounted for by the extensometer (Ref 22).

5. Conclusions

A preliminary experimental investigation on the mechanical performance of as FSW and FSW-SPF titanium 6Al-4V was conducted. For the first time, it is shown that the combination of friction stir welding and superplastic forming is a viable option for producing near-net shape components. Tensile properties are degraded, however, they are much better than

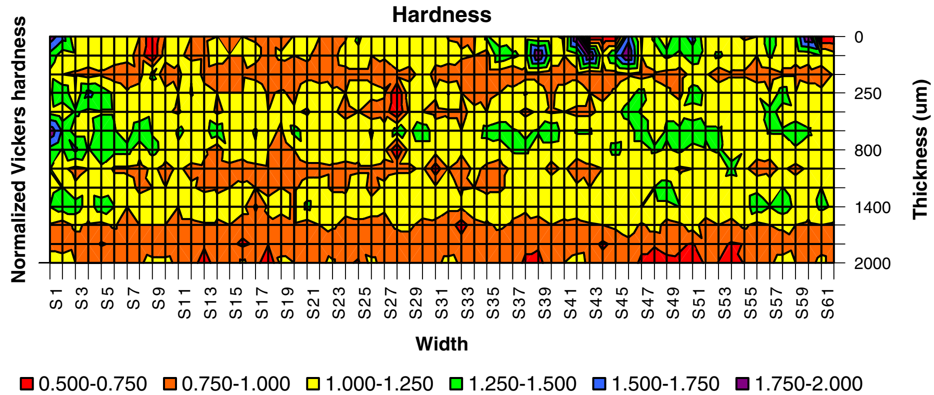

Fig. 12 Micro-hardness results (2-d mesh)

Table 1 Average tensile properties

Material type	Yield strength, MPa	Ultimate strength, MPa	Elongation, %
Fine grain	996	1035	5.0
Regular	1047	1086	5.2
SPF/FSW (regular)	1038	1090	2.7

Table 2 Results by SPF percentage

Pos.	Average SPF, %	Yield strength, MPa	Ultimate strength, MPa	Elongation, %
1	48.1	1013	1060	3.0
2	55.3	1044	1102	3.2
3	65.3	1043	1100	2.4
4	70.9	1052	1169	2.0

traditional fusion welding (Ref 23). In some cases, tensile strengths are within 8% of stock Ti 6Al-4V metal. With further optimization of the FSW process for titanium, even better results may be achieved and reported in future.

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References

1. M. Posada, J.P. Nguyen, D.R. Forrest, and J.J. DeLoach, Friction Stir Welding Advances Joining Technology, *AMPATIAC Q.*, 2003, **7**(3), p 13–20
2. R. Johnson and S. Kalle, Friction Stir Welding, *Mater. World*, 1999, **7**(12), p 751–753, http://www.azom.com/details.asp?ArticleID=1170
3. J.E. Gould, T.J. Lienert, and Z. Feng, Recent Developments in Friction Stir Welding. Society of Automotive Transactions, *J. Mater. Manuf.*, 1998, **107**, p 1093–1100
4. H.J. Liu, H. Fujii, M. Maeda, and K. Nogi, Tensile Properties and Fracture Locations of Friction-Stir-Welded Joints of 2017-T351 Aluminum Alloy, *J. Mater. Process. Technol.*, 2003, **142**, p 692–696
5. M. Peel, A. Steuwer, M. Preuss, and P.J. Withers, Microstructure, Mechanical Properties and Residual Stresses as a Function of Welding Speed in Aluminum AA5083 Friction Stir Welds, *Acta Mater.*, 2003, **51**, p 4791–4801
6. R.W. Fonda, J.F. Bingert, and C.J. Colligan, Development of Grain Structure During Friction Stir Welding, *Scr. Mater.*, 2004, **51**, p 243–248
7. G. Liu, L.E. Murr, C.-S. Niou, J.C. McClure, and F.R. Vega, Microstructural Aspects of the Friction-Stir Welding of 6061-T6 Aluminum, *Scr. Mater.*, 1997, **37**(3), p 355–361
8. C.G. Rhodes, M.W. Mahoney, W.H. Bingel, R.A. Spurling, and C.C. Bampton, Effects of Friction Stir Welding on Microstructure of 7075 Aluminum, *Scr. Mater.*, 1997, **36**(1), p 69–75
9. A.P. Reynolds, E. Hood, and W. Tang, Texture in Friction Stir Welds of Ti Metal 21S, *Scr. Mater.*, 2005, **52**, p 491–494
10. T. Trapp, E. Helder, and P.R. Subramanian, *FSW of Titanium Alloys for Aircraft Engine Components*, TMS Friction Stir Welding and Processing II, 2003, p 173–178
11. A.J. Ramirez and M.C. Juhas, Microstructural Evolution in Ti-6Al-4V Friction Stir Welds, *Mater. Sci. Forum*, 2003, **426–432**, p 2999–3004
12. W. Lee, C.Y. Lee, W. Chang, Y. Yeon, and S. Jung, Microstructural Investigation of Friction Stir Welded Pure Titanium, *Mater. Lett.*, 2005, **59**(26), p 3315–3318
13. O.A. Kaibyshev, Fundamental Aspects of Superplastic Deformation, *Mater. Sci. Eng.*, 2002, **A324**, p 96–102
14. H.G. Salem, A.P. Reynolds, and J.S. Lyons, Microstructure and Retention of Superplasticity of Friction Stir Welded Superplastic 2095 Sheet, *Scr. Mater.*, 2002, **46**, p 337–342
15. R.S. Misra and Z.Y. Ma, Friction Stir Welding and Processing, *Mater. Sci. Eng. Rep.*, 2005, **R50**, p 1–78
16. E.J. Klock-Mccook, "Characterization of Friction Stir Welded and Superplastic Formed Titanium Joints," MS Thesis, University of Washington, Dec 2005
17. P.D. Edwards, MS Thesis, University of Washington, June 2006
18. F. Pitt and M. Ramulu, The Effect of Simulated SPF Exposure and Post Processing on the Tensile Properties of 3 Titanium Alloys. *Proceedings from International Symposium on Superplasticity and Superplastic Forming Technology*, 2002, p 101–110 (ASM Publ)
19. F. Pitt and M. Ramulu, Influence of Grain size and Microstructure on Oxidation Rates in Titanium Alloy, Ti-6Al-4V Under Superplastic Forming Conditions, *J. Mater. Eng. Perform.*, 2004, **13**(6), p 727–734
20. F. Pitt, "Effect of Simulated Superplastic Forming Processing on the Mechanical Properties of Three Titanium Alloys," PhD dissertation, University of Washington, January 2005
21. C. Wang and G. Hou, A Study on Post-SPF Mechanical Characteristics of TI-6Al-4V Alloy, *Mater. Sci. Forum*, 1994, **170–172**, p 213–218
22. G.E. Dieter, *Mechanical Behavior of Materials Under Tension*, Metals Hand Book, vol. 8 Mechanical Testing, American Society for Metals, 1985, p 20–27
23. Material Property Data, January 17, 2006, http://www.matweb.com/search/SpecificMaterial.asp?bassnum=MTP642