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FRICION STIR PROCESSING OF LASER METAL DEPOSITED TI-6AL-4V

by

ROMY FRANCIS

A THESIS

Presented to the Faculty of the Graduate School of the

MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE IN MANUFACTURING ENGINEERING

2009

Approved by:

Dr. Frank Liou, Advisor
Dr. Joseph W. Newkirk
Dr. Ashok Midha

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PUBLICATION THESIS OPTION

This thesis consists of the following two articles:

Pages 2-29 are intended for submission to *Scripta Materialia*

Pages 30-53 are intended for submission to the *Journal of Manufacturing Processes*

ABSTRACT

Laser metal deposition (LMD) can form near net shaped components for aerospace applications. Ti-6Al-4V is a widely used aerospace alloy and fabrication of this alloy using LMD is gaining popularity. Improved mechanical properties are always a requirement in aerospace alloys and research is constantly being conducted to improve the properties of components formed from this alloy. Friction Stir Processing (FSP) in the past has been used as a method to change the surface properties of materials through microstructural modifications. The changes in properties can include hardness, corrosion resistance, fatigue strength, etc. The first section of this thesis is based on a similar concept to modify the surface properties of the laser deposited Ti-6Al-4V through FSP. Microstructural changes in the Ti-6Al-4V laser deposits due to FSP and subsequent laser deposition has been presented. The microstructure was characterized by the SEM and optical microscope. Variations in the microhardness inside the nugget region have also been discussed. The second section of the thesis proposes a methodology to create a fully forged, recrystallized structure by integrating the hybrid laser deposition system and FSP. Various FSP parameters investigated in attaining the same have been discussed. The effective nugget region which was left unaffected due to the subsequent single, double and triple layer laser deposition has also been presented.

ACKNOWLEDGMENTS

This research work is a result of some phenomenal help and support extended to me by many individuals at Missouri S&T. I would like to express my sincere gratitude towards my advisor Dr. Frank Liou and my co-advisor Dr. Joseph Newkirk for their continued guidance, encouragement and co-operation throughout my research work in the LAMP lab. It has been a pleasure working with them over the past two years and without their help this work would not have been possible. The research assistantship extended to me by Dr. Liou through the Manufacturing Engineering program is also greatly acknowledged.

I would also like to thank my committee member Dr. Ashok Midha for his time and advice granted to me during the research. I would like to express my sincere thanks to Dr. Greg Hilmas, Dr. Robert Landers and Dr. David Van Aken for allowing me to use their lab equipment. I sincerely thank all the members of the LAMP lab, especially Todd Sparks, Jianzhong Ruan, Ravi Philip and Rana Gunaratnam for helping me during the experiments and providing me with valuable suggestions which have been very critical in completing my research work. I would also like to thank Anand, Nilesh and Syamala for their time in helping me with the metallographic sample preparations. My sincere appreciation to Mr. Bob Hribar, Mr. Randall Lewis and Mr. Max Vath for the help they extended to me in the machine shop. I would also like to thank my roommates here at Rolla who have made the past two years memorable.

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INTRODUCTION

Layered manufacturing is gaining great importance in the aerospace industry due to its ability to create complex, near net shaped components within a shorter time frame and at a lower unit cost irrespective of the volume of the parts manufactured. The LAMP lab at Missouri S&T is capable of manufacturing components from aerospace alloys like Ti-6Al-4V using this unique technology. However, a need was realized for improving mechanical properties such as hardness and fatigue resistance on the surface of the materials manufactured using this technique. This research has focused on achieving this objective by the means of implementing Friction Stir Processing (FSP) as a surface modification technique by integrating it with the hybrid laser deposition system at LAMP. The work presented in this thesis comprises two sections. The first section provides details regarding the microstructural changes taking place in the laser deposit due to FSP under various scenarios such as substrate weld, deposition over stir zone, etc. The second section focuses on proposing a methodology of manufacturing a fully forged structure by using both FSP and the hybrid deposition system.

PAPER I

MICROSTRUCTURAL EVOLUTION IN FRICTION STIR PROCESSED

Ti-6Al-4V LASER DEPOSITS

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ABSTRACT

Laser metal deposition (LMD) is widely used as a manufacturing technique to create components from metal powder for aerospace applications. The microstructure of components obtained from this process is highly dependent on deposition parameters such as laser power, scan speed, powder feed rate, etc. This work has integrated friction stir processing (FSP) with the hybrid deposition system at the Missouri S&T LAMP lab and investigated this combination as a method to modify the microstructure of laser deposited Ti-6Al-4V components. Friction stir processing has the advantage of producing a microstructure independent of deposition parameters. The microstructure thus produced is also highly refined, a quality known to improve the resistance to fatigue crack initiation. Extremely refined microstructure (on the order of 1 μm) was observed in the stir zone (SZ) of processed Ti-6Al-4V deposits. Nugget regions have been characterized based on the two different tools and under different FSP scenarios like substrate weld, stir over deposit, etc. It also addresses microstructural evolution due to subsequent multi-scan laser deposition over the stir zone. In addition, it presents the results of the microhardness tests performed in the nugget region and discusses tool wear.

Keywords: Laser deposition, Ti-6Al-4V, friction stir processing

1. INTRODUCTION

Laser metal deposition (LMD) is a rapid prototyping technique which is used for manufacturing complex near net shaped components. This technology utilizes fewer raw materials and takes less time in producing the final component. The part obtained after the deposition requires minimal machining and this is an added advantage of this process over the conventional manufacturing processes. The microstructure obtained through this process is dependent on the laser deposition parameters which include the laser power, laser scanning speed, powder feed rate, etc [1]. Typical deposition microstructure in the case of Ti-6Al-4V consists of a basketweave Widmanstätten α morphology and colony Widmanstätten α morphology inside a prior β grain, continuous α along prior β grains have also been observed [2]. The grains can range between 100 μ m-600 μ m. The lamellar structure results in better creep properties and higher fracture toughness [8] whereas the equiaxed structures resist fatigue crack initiation.

Friction Stir Processing (FSP) has been investigated as a method for surface modification to form refined microstructure at the surface of the Ti-6Al-4V components manufactured from this method. In the past, FSP has been used to improve mechanical properties like the low cycle fatigue properties, hardness, corrosion resistance, and resistance to crack initiation in Al, Ni, Fe, Cu and Ti alloys [3, 4, and 5]. FSP works on the principles of Friction Stir Welding (FSW) [7] patented by TWI, UK. The only difference being that in the case of FSP no weld is formed. The rotating non consumable tool made from a refractory metal consisting of a profiled pin and a shoulder plunges into the material until the tool shoulder makes contact with the work piece. Once the tool shoulder makes contact, the tool is traversed in X or Y direction based on the clamping

constraints of the work piece. The material under the shoulder is plastically deformed and is thermo-mechanically processed due to the compressive forces and the stirring action induced by the rotation of the tool. This deformation occurs with high strain rates and the process yields a highly refined microstructure. Research was conducted earlier in the LAMP (Laser Aided Manufacturing Processes) lab to evaluate methods to form work hardened and a re-crystallized layer of the LMD Ti-6Al-4V parts [2]. Depths of up to 1mm of work hardened layer were obtained by rotational burnishing and up to 10 μm were obtained by aggressive milling. Integration of FSP in the LAMP lab is a step further in this direction to improve the product properties. The cooling rate in the case of laser deposits is very high and hence FSP can also be used to eliminate the internal stress if any. Lack of fusion voids [18] caused at the substrate-deposit interface can also be addressed by FSP.

2. EXPERIMENTAL METHODOLOGY

2.1. EQUIPMENT AND DEPOSITION PROCESS OVERVIEW

Friction stir processing (FSP) was performed using the FADAL 3016 CNC which is part of the multi axis hybrid laser deposition system at the Missouri S&T LAMP lab. The 1kW Nuvyonx diode laser (808 nm wavelength) forms the second part of the hybrid laser deposition system. The laser system consists of the laser cladding head, focusing optics, and powder delivering hoses which are attached to the Z axis of the CNC. Atomized metal powder is delivered from a powder feeder to the laser melt pool on the substrate while the X-Y axes of the CNC moves as per the G and M codes fed to it via the control system to deposit the 2D cross-section of the part. When one macro layer is deposited, the Z axis moves up by a predetermined height to refocus the laser to form a melt pool on the previously deposited layer and follow the build pattern for deposition. The approximate bead width in case of deposition with Ti-6Al-4V is 3mm. A 15 % overlap of the tracks was maintained during deposition. The three laser deposits, approximately 47mm x 26mm x 10mm in dimension were then deposited on a 50mm x 108mm x 9.5mm Ti 6Al-4V substrate. The laser deposition parameters for these deposits were not known since they were completed at an earlier date for another study. The deposition parameters of these deposits were not expected to significantly affect FSP. Ti-6Al-4V powder was supplied by Accumet Materials Co and was sized to -100 +325 mesh.

2.2. FRICTION STIR PROCESSING

Two different stepped FSP tools manufactured from Densimet-176 were used for this study. The tool shoulder did not have any features and no lead angle was used during

the run. Each tool consisted of a stepped pin profile resembling an inverted wedding cake. Each step acted as a shoulder and aided in heat buildup and plastic deformation. The step diameters were 2.8mm, 3.5mm, 4.5mm and the tool shoulder diameter was 7.6 mm. The steps were 0.5mm deep in case of Tool-1 and 1mm in case of Tool-2 (Figure 1).

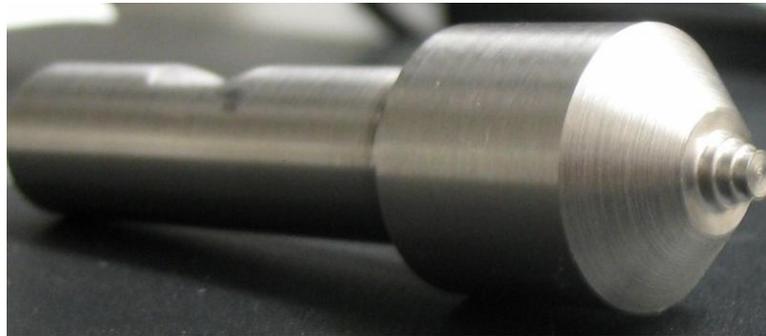


Figure 1. Stepped pin profile Tool-2

An atmospherically sealed argon gas-tight enclosure was used to prevent oxidation of the tool and Ti 6Al-4V deposit during FSP. Multiple trials were previously conducted with each of the tools to optimize the stir parameters and obtain defect free stir surfaces. Tool traverse feed of 50.8 mm/min (2 IPM) yielded the best stir surface finish and it also maintained the tool integrity. It was observed that the Tool-1 required approximately 10% higher RPM than Tool-2 which was being operated at 275 RPM to attain the intended plunge. This was due to the fact that only limited heat buildup was obtained with a shorter pin at the same parameters. This meant that even though Ti-6Al-

4V had a low thermal conductivity it was high enough to dissipate the thermal energy into the surrounding material requiring higher RPM for attaining the plunge and subsequent plastic deformation. The processing parameters used for the experiment have been summarized in Table 1.

Table 1 Friction stir processing parameters

Run ID	Plunge Feed	Traverse Feed	RPM
T1-103007.1 †	1.27 mm/min	50.8 mm/min	300
T2-102907.1	1.27 mm/min	50.8 mm/min	275
T1-103007.2	1.27 mm/min	50.8 mm/min	300
T2-102907.2	1.27 mm/min	50.8 mm/min	275
T1-103007.3	1.27 mm/min	50.8 mm/min	300
T2-102907.3	1.27 mm/min	50.8 mm/min	275

† All the stir runs have been named in the following manner (Type of tool) - (Month) (Date) (Year). (Run serial No: for that day)

Three sets of experiments were performed with each tool to evaluate the effects of FSP on the laser deposits. The first set comprised of laying one track of stir each with both the tools side by side on the same laser deposit. The second set of experiments were performed to study the effect of laser interaction with the stir zone (SZ). Single, double and triple pass of single track laser deposition was performed on top of the stir region in addition to the FSP of laser deposit. A third set of experiment consisted of stirring through the deposit into the substrate. This sample was obtained by milling down the existing laser deposit to leave approximately 1 mm of deposit on the substrate.

2.3. SAMPLE PREPARATION

The stirred samples were cross-sectioned perpendicular to the stir direction using resin bonded silicon carbide rotary blades. Samples were mounted using Diallyl Phthalate epoxy resin. Grinding and polishing was performed on a LECO Spectrum System 1000 automatic polisher. Blue, green and red cameo platinum plates corresponding to 120~180, 220~280 and 600 grit sandpaper were used for initial grinding. Two step polishing was performed using a 9 micron diamond solution and 0.05 micron colloidal silica on imperial cloth. The samples were rinsed thoroughly in water after the first polishing medium. To clean off the final polishing medium, the samples were placed in an ultrasonic bath of 95% ethyl alcohol for 20 minutes.

2.4. ANALYSIS

The Vickers microhardness tests were performed using the Struers Duramin 5 hardness testing machine with a load of 9.81N for 10 seconds. Three columns of 16 microhardness indents each were made on the samples. Three indent columns represented the advancing side of the nugget (N/4), centerline of the nugget (N/2) and the retreating side of the nugget (3N/4). The indents were separated by 250 microns to ensure that the readings were not biased.

For inspection, the samples were etched with modified Kroll's reagent (5ml HF, 22.5ml HNO₃, 22.5ml HCl) for approximately 15 seconds. The microstructure was later characterized using the Hitachi S570 scanning electron microscope, Hitachi S4700 Field emission microscope and the Nikon Epiphot 300 optical microscope. Backscattered compo mode was used for imaging in the SEM.

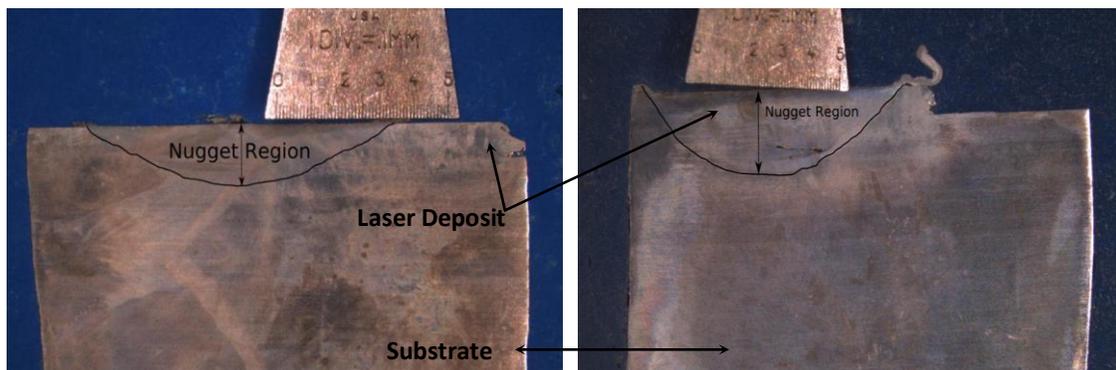
3. RESULTS AND DISCUSSION

3.1. MICROSTRUCTURAL EVOLUTION

3.1.1. Substrate welding experiment - stir zone. The nugget cross-section was shallower in the case of T1-103007.3 compared to T2-102907.3 (Figure 2 a, b) since a shorter tool was used. In both of these cases the nugget cross-section was observed to be nearly parabolic. The overall nugget cross-section consisting of the SZ, TMAZ and HAZ was outlined with an image editing software after etching the samples with modified Kroll's reagent. The observed depth at the center of the nugget (N/2) has been summarized in Table 2. In both cases, the FSP nugget region extended into the substrate through the laser deposit as per the requirements of the experiment.

Table 2 Substrate weld nugget depths

Run No:	Avg FSP Nugget depth (μm)
T1-103007.3	1670 \pm 25
T2-102907.3	2500 \pm 20



(a) Run no: T1-103007.3 substrate welding (b) Run no: T2-102907.3 substrate welding

Figure 2.

The temperature profile obtained from the optical pyrometer aimed at the tool shank slightly above the shoulder indicated that the tool shank temperature did not exceed 815°C which strongly indicates that the processing conditions existing underneath the tool shoulder would have been below Beta transus (995-1010 °C). The equiaxed microstructure of the substrate approx 16µm (Figure 3) and the Widmanstätten, lamellar morphology with $\alpha+\beta$ laths in the prior β grains (Figure 4) of the deposit region was transformed into highly refined, re-crystallized α microstructure (Figure 5) which had a diameter of around $0.80 \pm 0.08 \mu\text{m}$. The grain size was measured by the linear intercept method as per ASTM 112[12]. Rubal et.al [8] observed a similar morphology in the SZ when sub transus FSP was performed on near alpha Ti-5111 alloy. FSP studies on investment cast Ti-6Al-4V conducted by Pilchak et al. [4, 11] also showed equiaxed α microstructure in the sub transus FSP SZ. The microstructure was homogenized throughout the SZ converting the parent microstructures to equiaxed α .

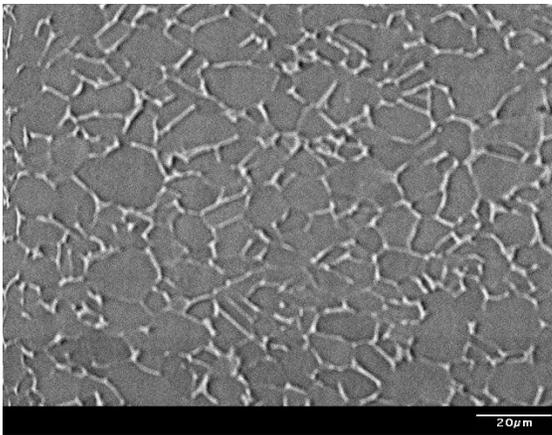


Figure 3. Equiaxed substrate microstructure

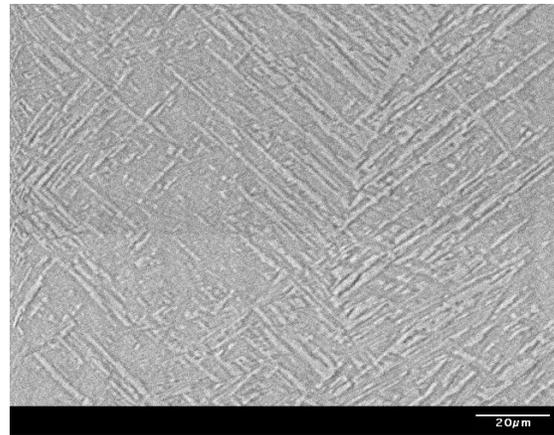


Figure 4. Basketweave structure

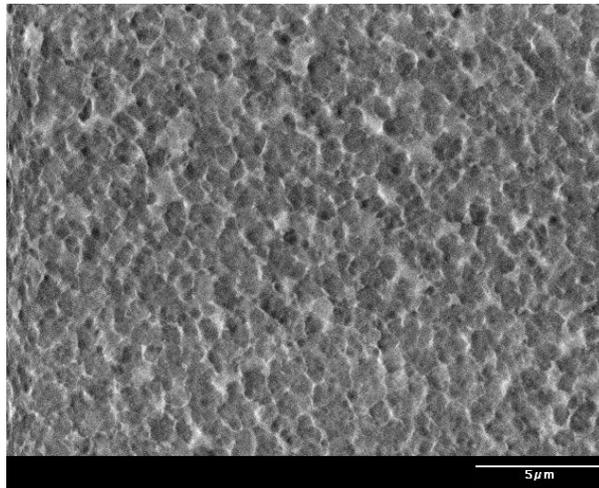


Figure 5. Equiaxed α grains

Ramirez et al. [3] observed that the SZ microstructure was dependent on the processing conditions namely tool traverse feed, tool RPM, forge force, tool design, shoulder features, etc., and not on the starting base microstructure. The SZ microstructure observation is in agreement with that finding. Although stir no: T1-103007.3 was performed at a higher RPM of 300, the SZ microstructure observed was similar to the T2-102907.3 indicating that the processing conditions during that run were also sub transus. Pilchak et al. [10] reported that if the material is being worked upon by the tool during its cooling from the β transus then the dislocation density in the subsequently formed equiaxed α would be high.

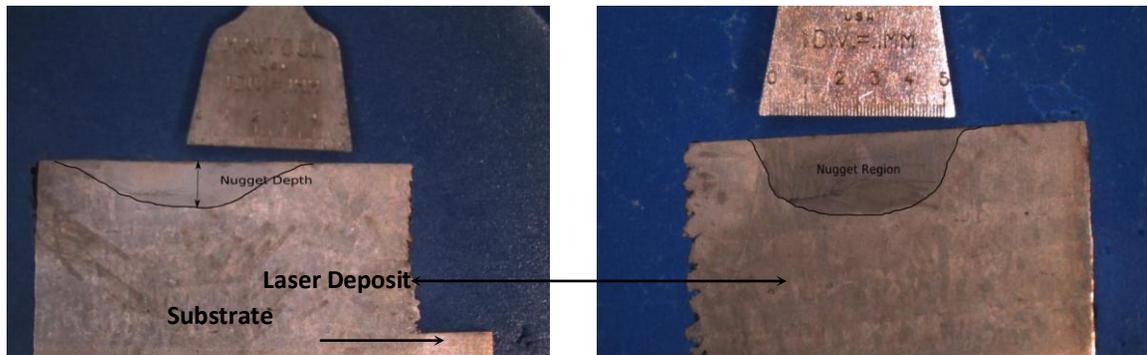
3.1.2. Stir over laser deposit – stir zone. The etched nugget cross-section for both T1-103007.1, T2-102907.1 (Figure 6. a, b) was not nearly as parabolic as observed for the substrate weld experiment. The reason for this could be that the difference in the BM (base material) caused different thermal cycles and heat dissipation patterns. The

bottom of the nugget region was observed to be much flatter. The SZ in case of this particular experiment also showed highly refined grains to the order of 1 micron. The depth at the center of the nugget region has been summarized in Table 3.

Table 3 Stir over deposit nugget depths

Run No:	Avg FSP Nugget depth (μm)
T1-103007.1	1600 \pm 23
T2-102907.1	2300 \pm 19

Although the tool processed the regions totally inside the laser deposit, the resulting microstructure consisted of equiaxed α grains. As discussed earlier, the microstructure of the laser deposit is primarily lamellar. SZ microstructure observation is in agreement with Peters et al. [14] who reported that the diameter of the recrystallized α grains in the thermomechanically processed bulk Ti-6Al-4V generally correspond to the width of the α lamellae in the BM. In this case the BM being the laser deposit, the α lamellae is to the order of \sim 1-2 μm . The temperature history showed that the tool shanks did not experience temperatures more than 820 °C.



(a) Stir over deposit run no: T1-103007.1 (b) Stir over deposit run no: T2-102907.1

Figure 6.

3.1.3. Transition zone (TZ) / (TMAZ) - substrate welding. Zhang et al. [16] reported a sharp boundary (SB) with no TMAZ in FSW of Ti-6Al-4V plates. It was reported that the deformation characteristics of TMAZ may have been masked by the subsequent phase transformations. Ramirez et al. [3] observed an extremely narrow TMAZ ($\sim 10\mu\text{m}$) zone. However extensive presence of equiaxed α was observed in this region. TZ which is analogous to this region was proposed by Pilchak et al. [10] as the region between the HAZ and the SZ which experiences measurable strain, but where the strain induced temperature gradient was insufficient to cause recrystallization as in SZ. However, a gradual change from the BM to SZ microstructure was observed in the substrate weld experiment. The microstructure of the BM is a beta annealed structure. The zone observed was approximately 70-80 μm wide (Figure 7).

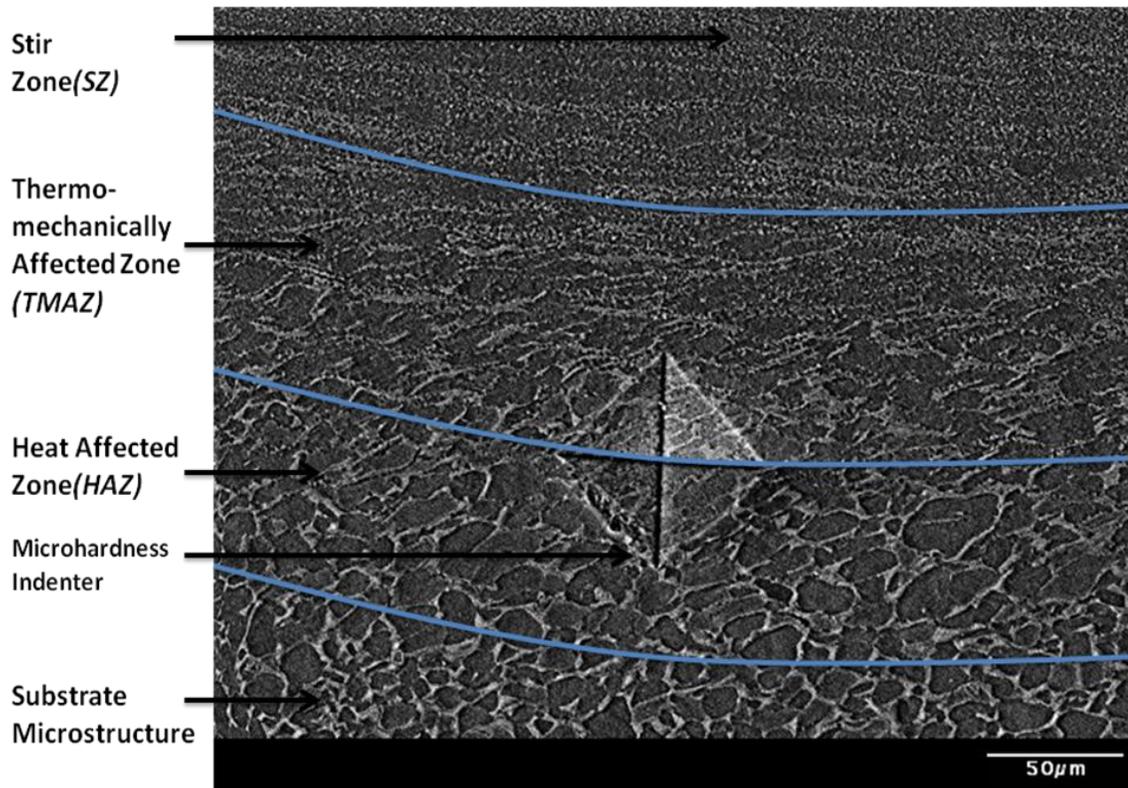


Figure 7. Nugget morphologies - T2-102907.3

This could be explained due to the fact that stirring deformation imparted from the SZ was primarily being resisted by the β annealed microstructure of the substrate. SZ microstructure, which was being formed as a result of dynamic recrystallization (DRX) from two different starting microstructures, would have caused a thermal conductivity gradient between the SZ and BM leading to a wider TZ. The TZ microstructure was observed to consist of deformed α/β lamellae and also equiaxed α (Figure 8). These grains were of the order of $1\mu\text{m}$.

Ramirez, Pilchak et al. [3, 10] observed similar morphologies in the TZ/TMAZ which was explained by lamellar α recrystallization which in the case of $\alpha+\beta$ is called α

globularization [13]. Seshacharyulu[13] et al. developed a microstructural deformation mechanism map for Ti 6Al-4V and based on that the existing strain rates in this region should be between 10^{-3} s^{-1} to 10^{-2} s^{-1} since the temperature at the SZ boundary is expected to be between 820° c and β transus.

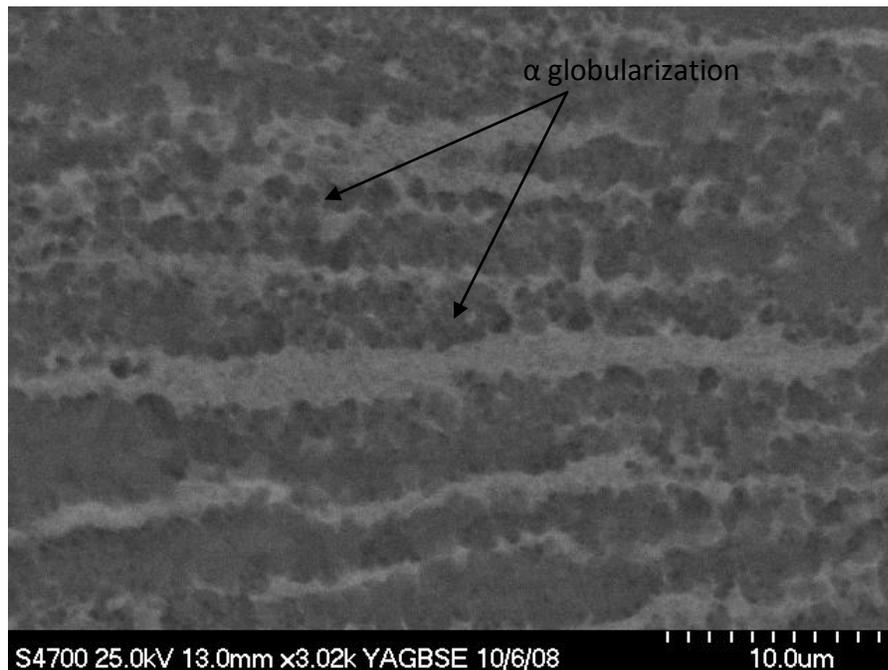


Figure 8. Equiaxed α structure along with deformed lamellae in TMAZ

3.1.4. TZ/TMAZ - Stir over laser deposit. TZ observed in this case was vastly different from the one which was observed in the substrate weld experiment. The TZ existed (Figure 9) but was only of the order of $25\text{-}30\mu$ as compared to almost $70\mu\text{m}$ which existed in the substrate weld. The shearing mechanism from the adjacent SZ could be related to this observation of abrupt change in the microstructure. Observations made

in TZ of the FSP laser deposit is in agreement with Pilchak et al. [10] who reported that the morphology of TZ is a function of lamellar α orientation of the adjacent BM, depth from surface and its location (AS/RS). Moreover it is clear with observations from TZ in both the substrate weld experiments that the TZ microstructure is more dependent on the BM microstructure.

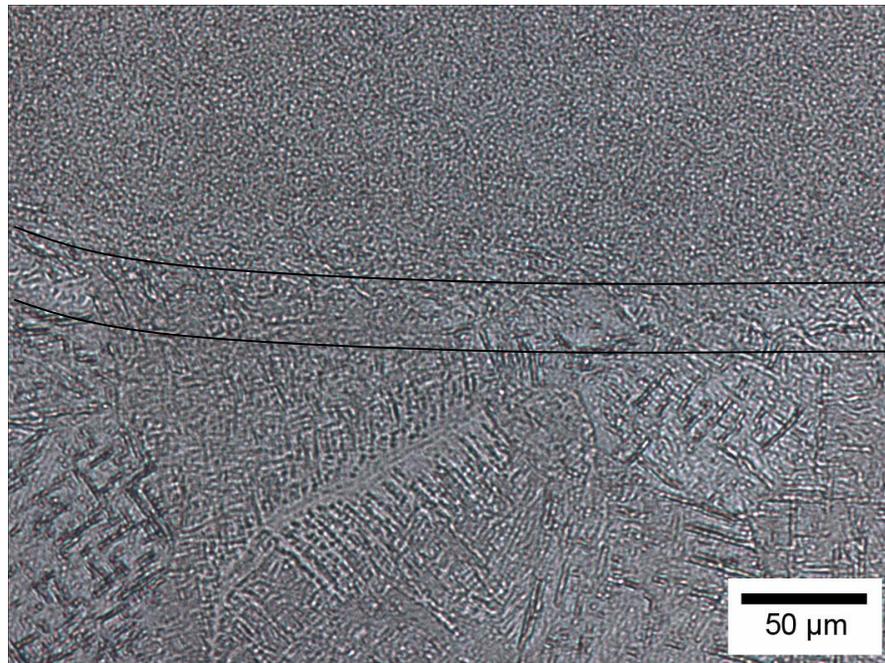


Figure 9. Narrow TZ on AS of the FSP laser deposit

3.1.5. Microstructural evolution in the deposit over stir experiments. Regions with varying microstructures were observed when cross-sections from the laser deposition over the stir zone were studied. These regions included the deposit dilution zone, laser deposition HAZ, stir zone (SZ), transition zone (TZ), deposit. The SZ and TZ

morphology did not differ from the previously described microstructures for the FSP over laser deposits.

The heat source from the laser would be primarily utilized in (a) melting the powder which is being deposited and (b) re-melting the already deposited layer (in this case it would be the FSP layer). It is clear from Figure 10 that the first layer of the deposit over the SZ consisted of large equiaxed grains to the order of 200 μm .

The HAZ from subsequent laser deposition over the FSP nugget showed a grain size gradient. Decreasing equiaxed grain size was observed as the depth increased from the surface of the stir. It was observed that the grains in the dilution zone were approximately 250 μm wide. An average grain size of 50 μm was observed at a depth 250 μm below the dilution zone. This was followed by a further decrease in grain size to 25 μm over the next 250 μm . More heat was absorbed by the grains closer to surface and the remaining heat aided in the grain growth of the equiaxed α in the nugget which was formed from FSP.

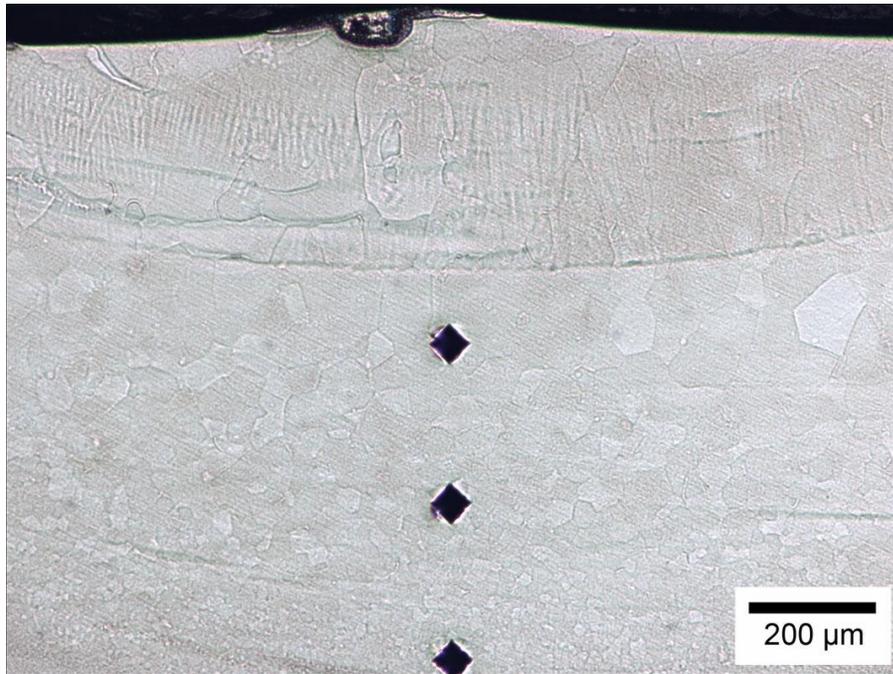


Figure 10. Center of the single track-single pass deposition over run no: T1-103007.2

Backscattered SEM imaging of an area very close to the deposit in run T2-102907.2 with a triple pass of laser revealed the grain morphology as shown in Figure 11. The morphology clearly shows a fine basketweave microstructure inside a prior β . It is evident that this region experienced temperatures above the β transus.

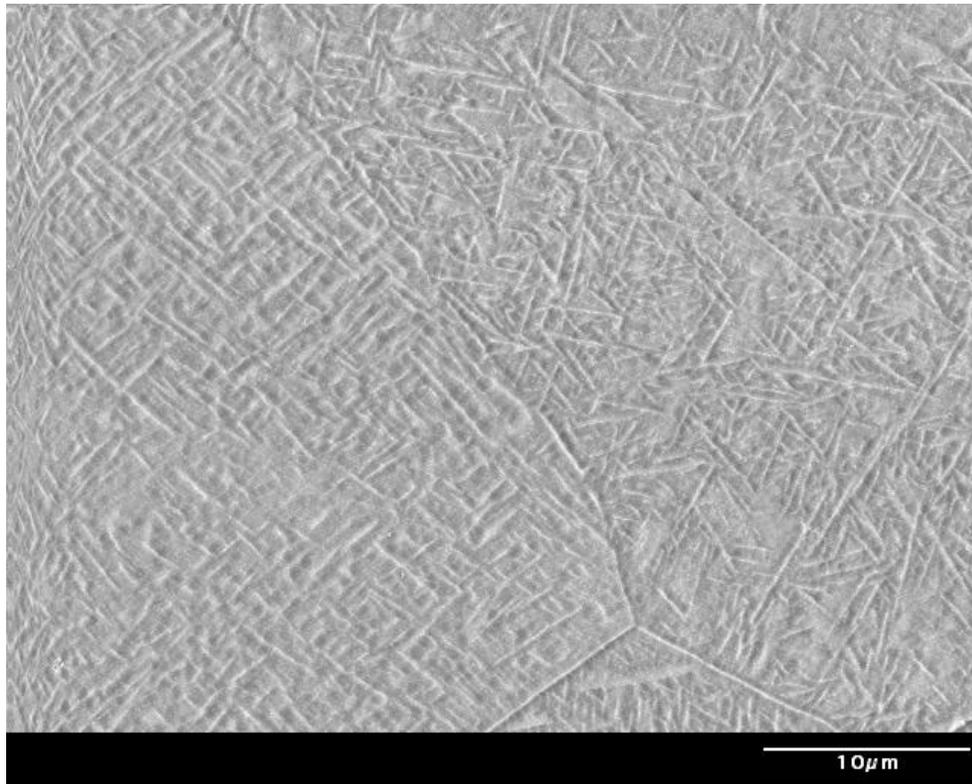


Figure 11. Fine basket weave structure close to the deposit over the stir region

It was also observed that in all the deposition over the SZ (single, double and triple pass), a zone of approximately 100μm wide existed around the HAZ. This interface consisted of extremely fine grains and it was evident that these grains did not experience temperatures above β transus. The same has been shown in the following Figures 12 a and b.

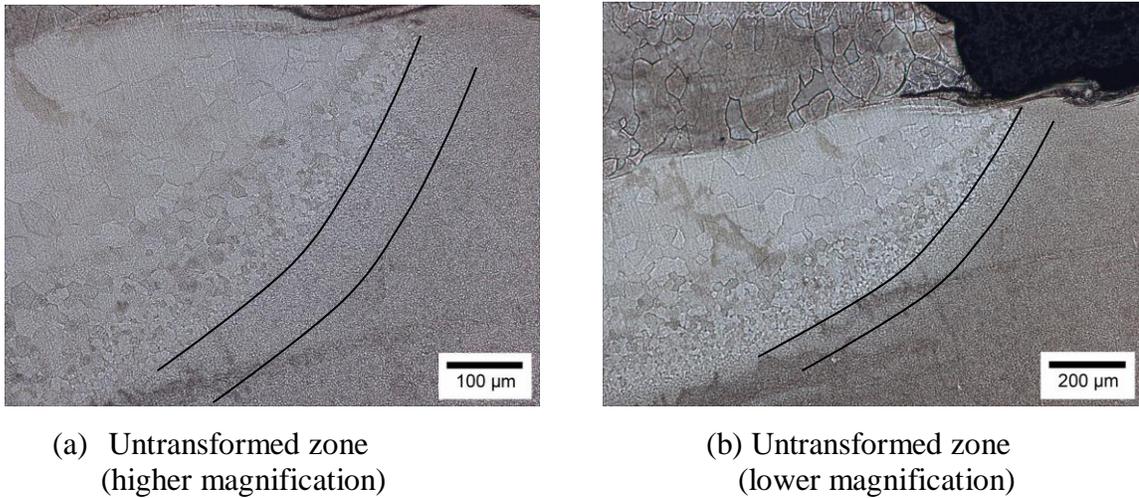


Figure. 12

3.2. MICROHARDNESS

3.2.1. Substrate weld microhardness. Microhardness test data of the nugget center (N/2) of runs: T1-103007.3 and T2-102907.3 revealed that the hardness had increased noticeably in both the runs in the nugget region. Lower RPM stirs made with Tool-2 imparted more hardness to the SZ compared to Tool-1. Similar trends were observed when the microhardness data was compared on the advancing side (N/4) and the retreating side (3N/4). It is worthwhile to note that the hardness kept decreasing as a function of depth which can be explained as more plastic strain was imparted by the tool shoulder at the top of the nugget compared to the remaining volume inducing more hardness near the top of the nugget.

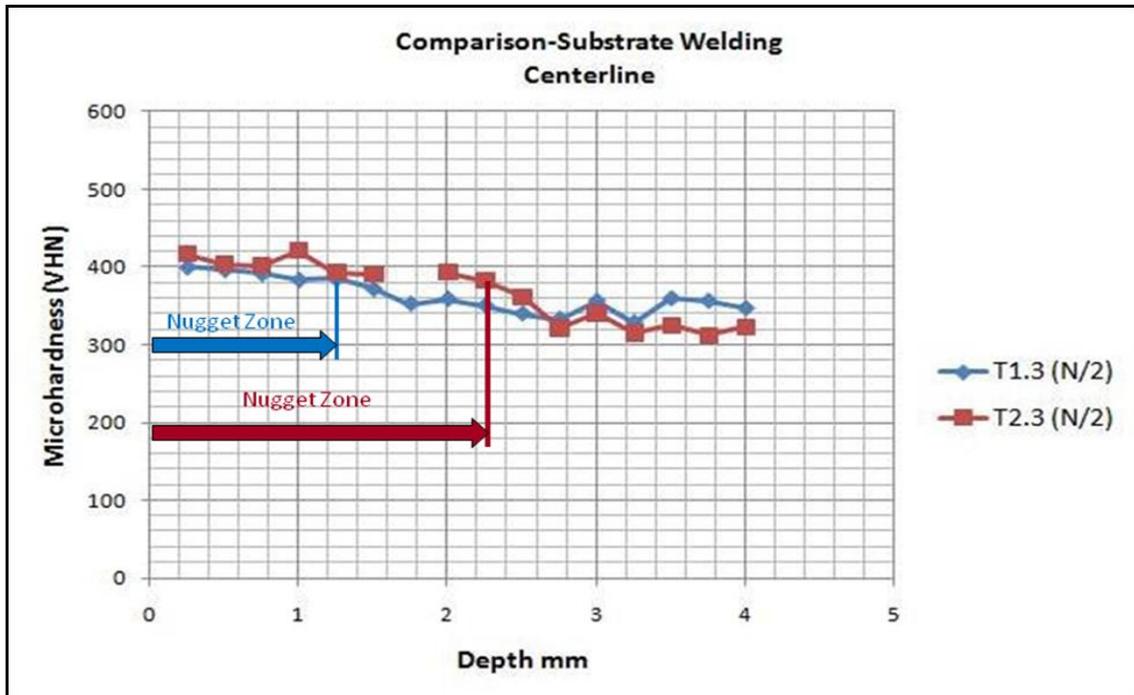


Figure 13. Microhardness data for substrate welding experiment

3.2.2. Stir over deposit microhardness. Microhardness test data for the stir over deposit (Runs: T1-103007.1 and T2-102907.1) along N/2 of the SZ revealed that the hardness had increased noticeably for the run with Tool-2 (Figure 14) but not as much for the run with Tool-1. Similar trends were observed when the microhardness data was compared at N/4 and 3N/4. As mentioned earlier this could be possible due to the higher dislocation density which could have been created due to processing at lower temperatures. In both the experiments it was observed that hardness profiles in the laser deposit and substrate region beyond the nugget closely followed each other indicating that deposit hardness was uniform.

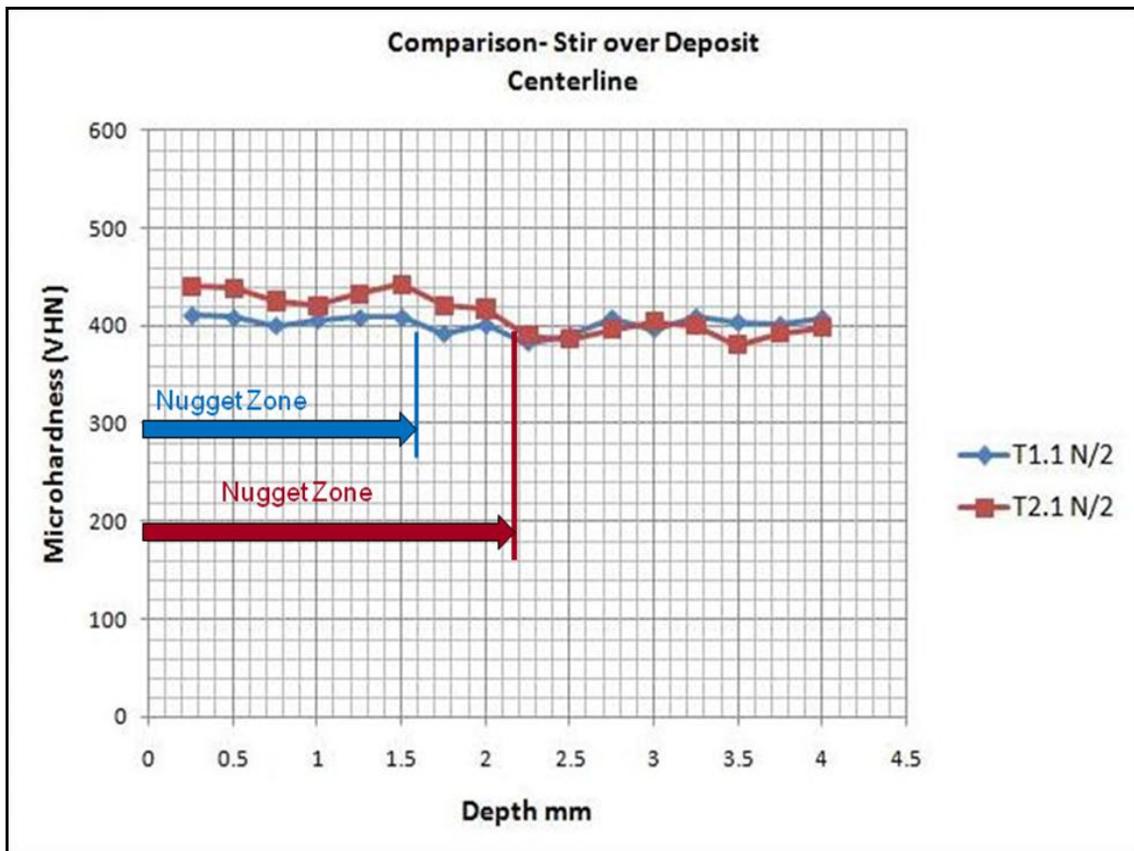


Figure 14. Microhardness data for stir over deposit experiment

3.2.3. Deposit over stir microhardness. Increased hardness was observed in the HAZ formed due to subsequent laser deposition over the SZ (Figure 15). This was true for all the nugget regions where the laser interacted. It was also noted that the deposit dilution zone was softer than the HAZ from subsequent laser deposition and this is the reason for the initial surge in hardness. In case of double pass laser deposition it was observed that hardness values decrease as a function of depth before stabilizing in the stir zone which was not affected by the laser HAZ.

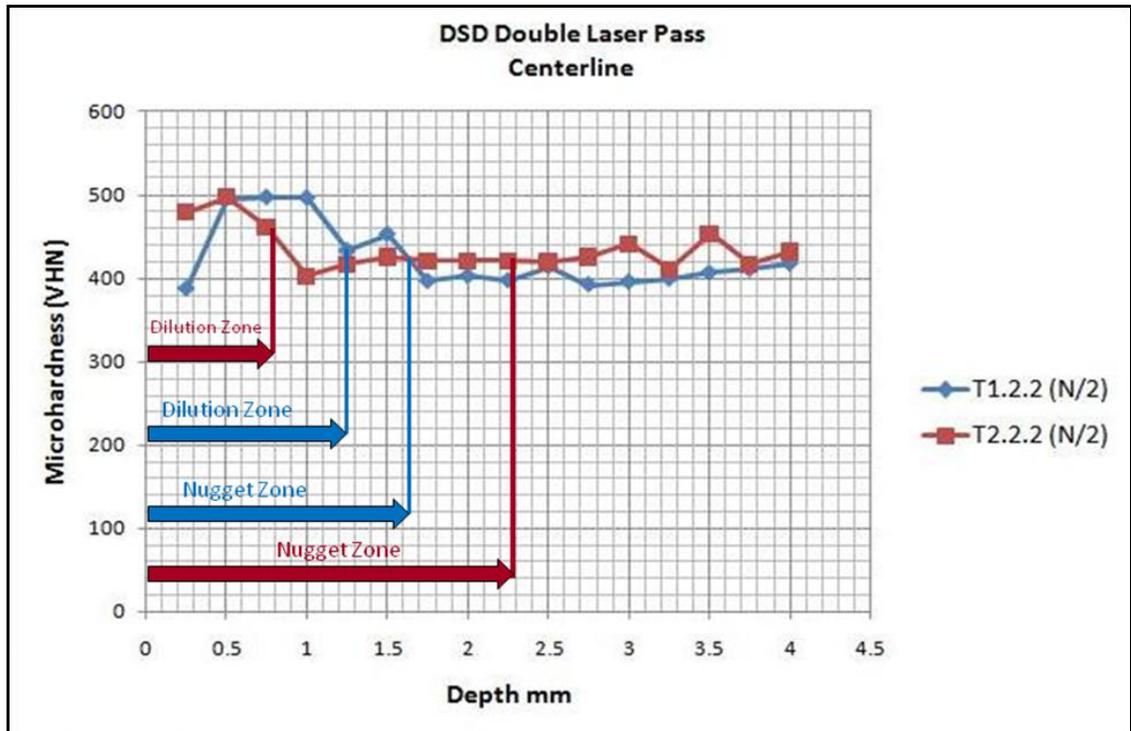


Figure 15. Microhardness data for deposit over stir experiment

3.3 TOOL WEAR AND PROCESSING DEFECTS

Pilchak et al [17] reported sub micron tungsten tool inclusions from the W-25% Re tool during FSP of the investment cast, hot isostatically pressed Ti-6Al-4V. W is a beta stabilizer and being higher in the atomic number shows up bright in the SEM images (Figure 16). Densimet-176, also a W based alloy was used for this experiment and a bright stripe was observed below the processing defect formed inside the nugget region. EDS analysis performed on this area confirmed the presence of W.

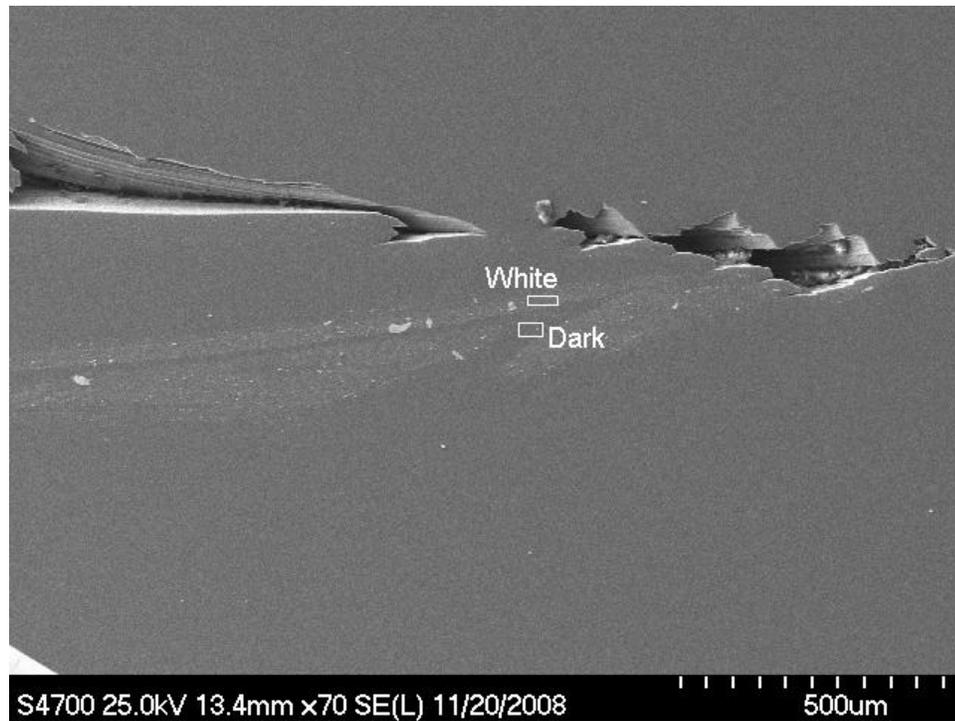


Figure 16. Processing defect observed in substrate weld experiment

This processing defect was observed ~ 1.9mm from the surface of the stir. A tool profile when superimposed on the nugget region revealed that the defect had formed in close proximity to 2nd-3rd shoulder interface. Processing voids could have been formed for a variety of reasons. One cause could be related to the laser deposits prepared with un-optimized deposition parameters. There were pores in the laser deposit which were revealed when the deposit cross-section was analyzed. These pores likely would have accumulated on the advancing side of the tool during processing resulting in a macro void. The lead angle on the tool helps in providing a downward forge force and the absence of this lead angle due to system constraints during this FSP experiment could have also contributed to this defect.

3.4 FATIGUE LIFE

Fatigue tests were not performed on the FSP laser deposit samples but certain predictions can be made about the fatigue life performance based on the microstructure and previous published work. M.Peters et al. [15] have reported that by reducing the α grain size from 12 μm ~15 μm to an equiaxed 1 μm ~2 μm , the Ti-6Al-4V alloy corresponded to about 25% increase in fatigue strengths at 10^7 cycles. Pilchak et al. [6] reported increased fatigue life and compressive yield strengths after the four point bend tests and micropillar compressive tests on FSP investment cast and HIP Ti-6Al-4V. It was observed from their study that the number of cycles to failure increased by a factor of 10 for the FSP samples in comparison to the base material. Although considerable scatter was observed in the results, the increase in fatigue strength is evident. It was concluded in their study that the improved fatigue life could have been attributed to the reduced slip length from several hundred micrometers of the α colony size to the α grain size which was around 1 μm . The equiaxed α microstructure showed a 12% increase in the compressive yield stress. The SZ microstructure found in this study pertaining to FSP of Ti-6Al-4V closely resembles the microstructure observed by Pilchak et al. and a similar fatigue response is expected.

4. CONCLUSION

Friction stir processing of the laser deposited Ti-6Al-4V deposits was performed and optimum processing parameters were obtained. The microstructure of the nugget regions obtained in the substrate weld, stir over deposit and deposit over stir experiments was presented. It was observed that FSP modifies the BM microstructure to a highly refined equiaxed α microstructure. Large equiaxed grains were observed in the experiment where subsequent deposition was carried over the stir. A decreasing grain size gradient existed in the HAZ formed due to subsequent laser deposition over the SZ. Presence of a band approximately 100 μ m wide consisting of untransformed grains which did not experience β transus was also observed in all the samples around the HAZ from laser interaction. Hardness imparted by Tool-2 after FSP was higher in comparison to Tool-1. It was also noted that the hardness in the HAZ from deposition was harder than the SZ nugget. Tool wear was observed during this process, EDS analysis of the nugget showed the presence of tungsten particles. Other studies indicate that the highly refined microstructure formed from FSP in Ti-6Al-4V has been able to increase the fatigue life by delaying the fatigue crack initiation. A similar performance is expected from the microstructure obtained in this study.

5. ACKNOWLEDGMENTS

This research was supported by the National Science Foundation grants DMI-9871185 and IIP-0637796, the grant from the U.S. Air Force Research Laboratory contract # FA8650-04-C-5704. The support from Boeing Phantom Works, Product Innovation and Engineering, LLC, Spartan Light Metal Products Inc, Missouri S&T Intelligent Systems Center, and Missouri S&T Manufacturing Engineering Program, is also greatly appreciated.

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PAPER II
**AN APPROACH TOWARDS BUILDING A FRICTION STIR PROCESSED
STRUCTURE USING THE HYBRID LASER DEPOSITION SYSTEM**

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ABSTRACT

This paper discusses the results of experiments carried out by integrating friction stir processing (FSP) with a layered manufacturing system. Optimum friction stir processing parameters like tool traverse feed, RPM, etc., were obtained for both the FSP tools used to process laser deposited Ti-6Al-4V. The methodology of arriving at the optimum parameters for better stirs under different experimental settings has been discussed. The extent of deformation due to FSP on a thin wall was also discussed. The effect of subsequent laser deposition on the stir zone was presented on the basis of depth of dilution zone in the stir zone nugget. It was observed that 1100 μ m-1300 μ m of highly refined microstructure was left unaffected even after laser deposition. A methodology was then proposed to use FSP as a layered manufacturing process to create a fully forged, recrystallized microstructure.

Keywords: Friction stir processing, Ti-6Al-4V, laser metal deposition, freeform fabrication

1. INTRODUCTION

Ti-6Al-4V is a commonly used aerospace alloy due to its excellent strength to weight ratio. However challenges exist in manufacturing components from this alloy by the traditional methodologies owing to the cost associated with the material lost in scrap. Innovative solid free form fabrication techniques have been constantly researched and developed for the last two decades in order to face the challenges of manufacturing in the aerospace industry. Arcella et al. [1] have described that using the laser aided manufacturing (LAM) process, near net shaped aerospace components can be created from Ti-6Al-4V alloy. This process can develop complex machining performs with high metallurgical integrity at a lower cost and lower lead times [2].

Friction stir welding and processing [3], a relatively new technique initially introduced to weld Al alloys and also to improve the microstructure is now being used for welding and processing Ti alloys. Recently Threadgill et al. [4] discussed friction stir welding being used as an additive manufacturing technique for Ti alloys. They demonstrated that a near net shaped, layered structure can be prepared by deposition using static shoulder friction stir welding (SSFW) [5] of Ti-6Al-4V. Romy et al. [6] recently used FSP to modify the laser deposited microstructure of Ti-6Al-4V alloy and were able to attain grains to the order of $1\mu\text{m}$. Pilchak et al. [7] have reported improvement in fatigue life of friction stir processed, investment cast Ti-6Al-4V owing to the refined microstructure.

The current study is aimed at proposing a methodology for preparing a fully forged, recrystallized, laser deposited structure with a layered manufacturing approach. This would be achieved by utilizing the capabilities of the multi axis hybrid deposition

system developed at the Missouri S&T for preparing the laser deposits and subsequently performing friction stir processing (FSP) using the same equipment on the deposit. This cycle of deposition, FSP and subsequent deposition would be repeated until the desired build height is attained.

2. EQUIPMENT OVERVIEW

The Hybrid laser deposition system at the Missouri S&T LAMP lab consists of a 1kW Nuvyonx diode laser (808 nm) and a 5 axis FADAL 3016 Vertical Machining Center. Atomized metal powder from a Bay State powder feeder is delivered to the laser melt pool while the substrate which is clamped on the rotary vice translates in the X-Y plane to form a layer representing a 2 dimensional slice of the part geometry to be deposited. The motion in the X-Y table of the CNC is governed by the codes which are generated by the process planning software [8]. This data is communicated to the CNC through a National Instruments real time system. After one layer of the deposit is made, Z axis translates upwards and starts depositing on the previously deposited layer and continues the build process to form uniform/non uniform layers of the deposit resulting in a near net shaped laser deposit. Once the geometry is deposited, the part is further processed by a milling tool to obtain the exact dimensions.

The Friction Stir Processing (FSP) tool is used in the same spindle which is used for milling. Thus the machine is capable of depositing, machining and performing FSP at the same time. FSP however is not performed on the rotary vice, instead on a vice which is mounted on the X-Y table. FSP generates excessive force in the X-Y plane due to the stirring action and the translational motion and hence a more robust vice was used to clamp the substrate.

3. PROCESS METHODOLOGY

A series of experiments were conducted to propose a methodology for building a FSP structure using the LAMP deposition process. Experiments were mainly conducted in four phases followed by the analysis of the samples. The flowchart for the process methodology has been shown in Figure 1.

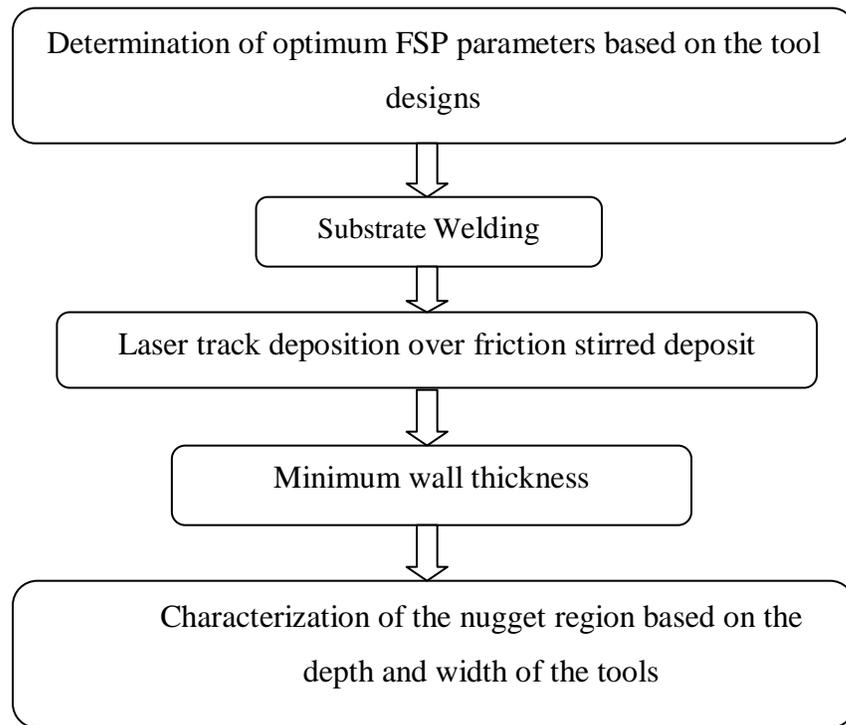


Figure 1. Process methodology flowchart

3.1. DETERMINATION OF OPTIMUM FSP PARAMETERS

3.1.1. Experimental phase-1. Two different FSP tools were designed from Densimet-176 which is Tungsten based alloy for performing the stirs. The tool shoulder did not consist of any features and no lead angle was used during the runs due to equipment limitations. The inverted wedding cake design was used for each tool pin

profile. Each step of the tool pin was designed so that it would act as a shoulder which would in turn help in heat buildup and aid plastic deformation so that better stirs would be obtained. The step diameters were 2.8mm, 3.5mm, 4.5mm with the shoulder diameter being 7.6 mm. Tool-1 total pin height was 1.5 mm while Tool-2 was 3 mm deep. All the stirs for the laser deposits were performed under an argon gas enclosure to prevent the oxidation of the tool and processed surface. The stirs were performed in position control mode. The experimental matrix for the initial set of experiments is summarized in Table 1 and the stirs obtained are shown in Figure 1.

All the deposits were prepared using Ti-6Al-4V powders which had a size range of -100 +325 mesh. The deposition was carried out at 780 watts laser power with 15% track overlap. The deposition geometries were thin walls (8mm) and thick walls (18mm). This was done to assess if the part geometry had influence on the process. The plunge was carried out at 0.021mm/s (0.05 IPM) and traverse feed for the tool was 0.211mm/s (0.5 IPM). Important conclusions were made regarding the FSP parameters from this study. It was understood that at a low RPM of 200 (Runs 2, 9-not shown in the images) it was difficult to attain penetration using both the tools. A higher RPM of 300 in Tool-2 caused the material to become very hot and swirl upwards resulting in improper stirs. Therefore the stirs for future experiments were performed between 250 and 300 RPM.

Table 1 Experimental phase-1

Run No:	Factor-1: Tool	Factor 2: Geometry	Factor 3: RPM
1	Tool-1	Thick	250
3	Tool-1	Thick	300
4	Tool-2	Thick	250
5	Tool-2	Thin	250
6	Tool-1	Thin	300
7	Tool-1	Thin	250
8	Tool-2	Thick	300
10	Tool-2	Thin	300



Figure 2. Stirs obtained from phase-1 experiment

3.1.2. Experimental phase-2. Higher feed rates were utilized for the next set of experiments performed with Tool-1 and Tool-2 on thin walls. The deposition parameters

for these laser deposits were not known. Different feed rates were used while keeping the RPM constant at 250. The plunge feed was maintained at 0.021 mm/s (0.05 IPM). The FSP parameters and measured surface roughness values have been tabulated in Table 2. Surface roughness is an important parameter because during the subsequent FSP wall buildup it is important to deposit on a surface that does not have voids.

Table 2 Phase 2 FSP parameters and surface roughness

Run ID	Traverse Feed	Surface roughness
T1-091207.1†	0.3175 mm/s (0.75 IPM)	Ra > 250 μ inches*, Very rough surface finish
T1-091207.2	0.423 mm/s (1.0 IPM)	Ra > 250 μ inches, Improved surface finish than T1-091207.1
T1-091207.3	0.635 mm/s (1.5 IPM)	Ra > 250 μ inches, Surface finish similar to T1-091207.1
T2-092107.2	0.423 mm/s (1.0 IPM)	Ra = 250 μ inches, Smoother surface finish for the initial 0.2 inches
T2-092107.3	0.635 mm/s (1.5 IPM)	Ra > 250 μ inches, surface voids originate at the center and then move towards advancing side of the stir
T2-092107.4	0.846 mm/s (2.0 IPM)	Ra = 154.30 μ inches, Smooth surface finish. Fine voids in advancing side
T2-092607.1	0.846 mm/s (2.0 IPM)	Ra = 120.36 μ inches, Good Surface finish. The surface gets rough towards center

*250μ inch is the upper limit of the surface measurement instrument used

† All the stir runs have been named as per the convention (Type of tool) - (Month) (Date) (Year). (Run serial No: for that day)

It was observed that higher RPMs yielded better surface finishes, but the surfaces obtained were still not defect free.

3.1.3. Experimental phase-3. Phase-3 of the experiment (Figure 3) was performed based on the data and expertise gained from performing FSP on previous Ti-6Al-4V laser deposits. These laser deposits were also prepared with 15% track overlap. 3 deposits were prepared and FSP tracks with both the tools were laid side by side on each of these deposits. Each of the 6 runs was performed at 0.846 mm/s (2 IPM) traverse feed and a plunge feed of 0.021 mm/s (0.05 IPM). The processing parameters and surface roughness obtained are presented in table 3.

Table 3 Phase 3 FSP parameters and surface roughness

Run ID	RPM	Surface Roughness
T1-103007.1	300	Ra=117 μ inches, Very good surface finish at the middle of the run
T2-102907.1	275	Ra=88 μ inches, Extremely smooth surface finish towards the end of the run
T1-103007.2	300	Ra=138 μ inches, Good surface finish, roughness increases towards the end of the run
T2-102907.2	275	Ra=218 μ inches, Surface finish becomes rough after the initial 0.5 inches
T1-103007.3	300	Ra=123 μ inches, Very good surface finish, fine voids on the AS
T2-102907.3	275	Ra=169 μ inches, Good surface finish for the initial 0.25 inch of the run

*250 μ inch is the upper limit of the surface measurement instrument used

It was observed that the processing conditions yielding better surface finish for Tool-1 existed close to 300 RPM and 0.846 mm/s (2 IPM) while processing thicker walls. The ideal conditions for Tool-2 existed around 275 RPM, 0.846 mm/s (2 IPM) while processing thick walls and around 250 RPM while processing thin walls at the same traverse speed.

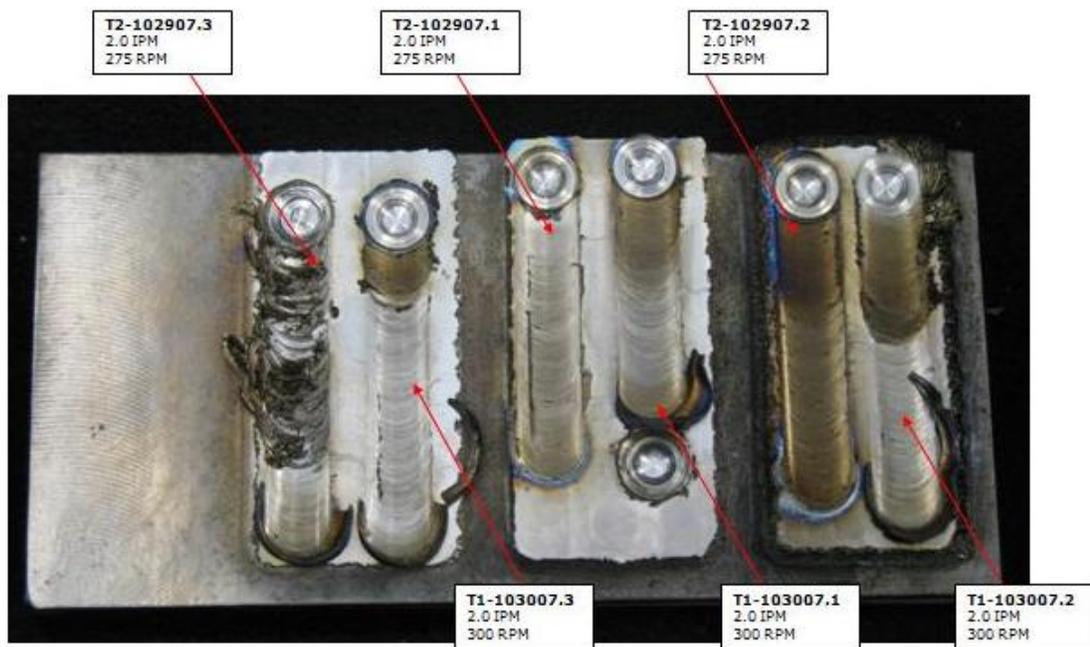


Figure 3. Phase 3 stir results

3.2. SUBSTRATE WELDING

Runs T1-103007.3 and T2-102907.3 were the substrate weld experiments. The laser deposit on the substrate was machined down to leave a thin layer of deposit. FSP was then performed so that the tool penetrates into the substrate through the deposit. The

thickness of the deposit left after milling was ~ 1 mm. A schematic of the substrate weld experiment is shown in Figure 4(a) and (b).

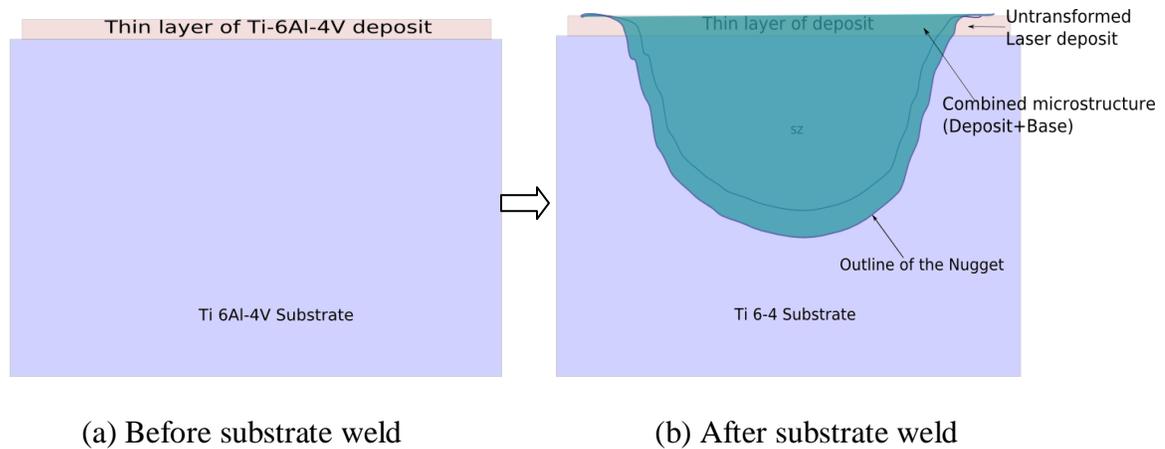


Figure 4.

This experiment was performed to understand if the deposit and the substrate microstructures merge into one single microstructure due to plastic deformation subsequently solving the issues arising from lack of fusion at the substrate and deposit interface [9]. It would also serve as a foundation for the friction stir processed structure build.

3.3. DEPOSITION OVER FRICTION STIRRED REGION

This experiment was performed as the next step in developing the methodology to build the FSP structure. Friction stir processed wall building would involve laser deposition over the stirred region as an intermediate step in attaining a fully forged

microstructure and it is imperative to understand how much stirred region of the nugget was left unaffected by the laser deposition. Figure 5 (a) and (b) show the schematic of the nugget region and the placement of the laser track over the stir tracks respectively.

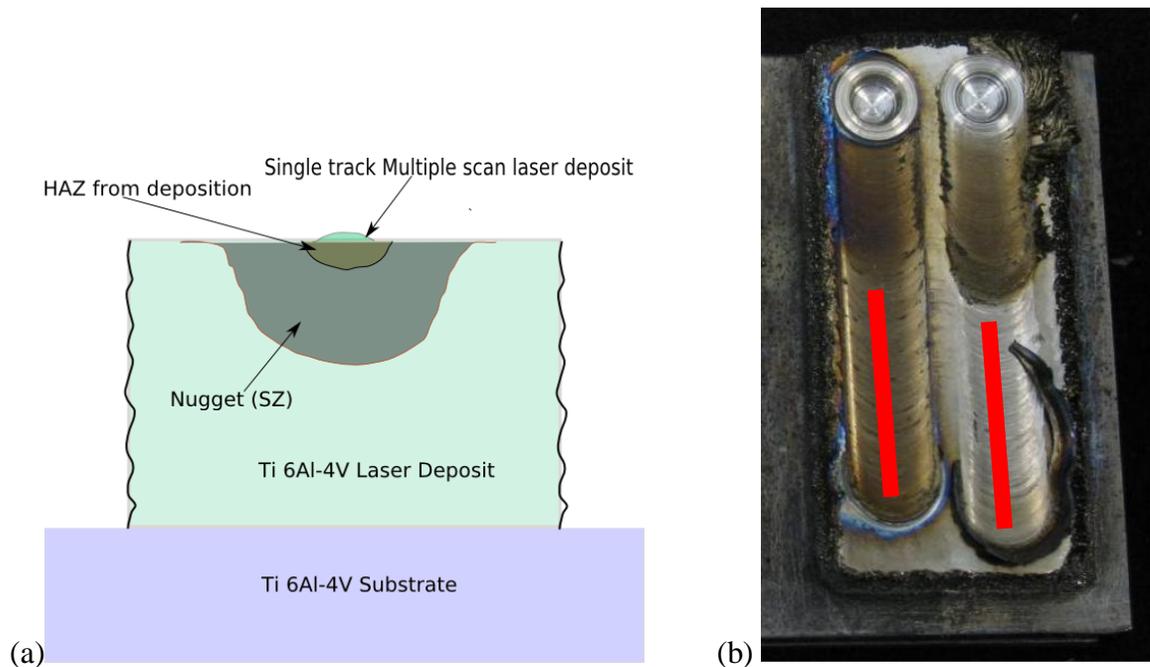


Figure 5. (a) Laser HAZ inside the stir nugget, (b) Track placement over the stir surfaces

A single track-single scan, double scan and triple scan deposit were made over the stirred region. The deposition was performed at 1kW laser power and 6.35mm (15 IPM) travel speed with Ti-6Al-4V powder. This parameter was chosen because it had yielded better deposits with Ti-6Al-4V and moreover maximum laser power of 1kW meant that the largest heat affected zone (HAZ) was being created in the stir nugget. Run numbers

T1-103007.2 and T2-102907.2 were used for this study. The samples obtained were sectioned, mounted on conductive Bakelite and analyzed after etching with modified Kroll's reagent. The outline of the nugget region formed was mapped out and the total nugget depth which included the stir zone (SZ), transition zone (TZ/TMAZ) and the heat affected zone (HAZ) was measured with image processing software.

3.4. MINIMUM WALL THICKNESS SCENARIO

Another important metric of the FSP wall building methodology would be to identify the minimum wall thickness that could be stirred. FSW is considered to be a low distortion process [4] however the process loads can vary significantly depending on the plunge feed, tool design, tool rotation speed and the material being processed. It was therefore important to quantify the distortion that would be observed while processing a thin wall of Ti-6Al-4V deposit. Figure 6 (a) and (b) shows direction and front view of stir no: T2-092607.1 which was performed after milling the deposited wall down to 7.8 mm (~0.31 inches) width. This was done so that any apparent distortion would be visible.

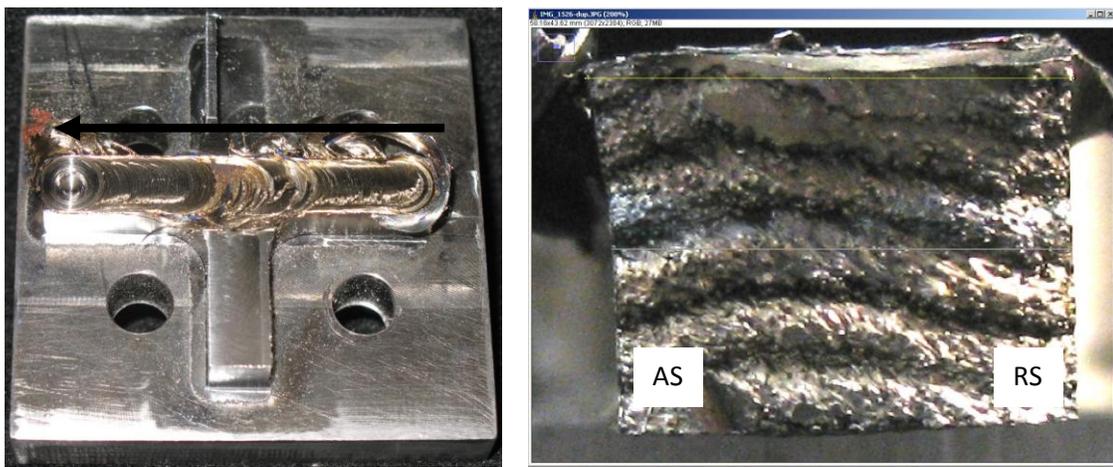


Figure 6. (a) Run no: T2-092607.1

(b) Front view of run no: T2-092607.1 deposit

Close observation of the front view of the run revealed that the wall had a mushrooming effect towards the top. There was a minimum of 5-6% distortion in the dimensions from base to top. Image J software was used for this study. It was noted that the wall had distorted on the advancing side (AS) of the run and that the retreating side (RS) of the deposit remained nearly unaltered. Arbogast et al. [10] reported slightly higher temperatures on the AS where the tangential velocity vector is in the same direction as of the forward traverse direction. This observation was made while performing FSW on Al. As the thermal conductivity of Ti-6Al-4V is not very high it could have aided heat buildup and subsequently assisted in the slight deformation of the wall on the AS. It is worthwhile noticing that the deformation was not observed in the FSP of the thin wall which was not milled down around the edges. Hence the minimum wall thickness for stirring with the process parameters being used would be 8mm.

4. RESULTS AND DISCUSSION

4.1. CHARACTERIZATION OF THE LASER HAZ IN THE NUGGET

The nugget depth obtained in the substrate weld and stir over laser deposit have been previously reported by Francis et al. [6]. In this paper, data pertaining to the laser interaction with the stir zone have been included. Figures 7, 8 and 9 show the macro cross-sections of the experiments with single, double and triple scans of the laser respectively. The nugget depth and the HAZ resulting from the single double and triple laser scans have also been reported in Tables 4, 5 and 6 respectively.

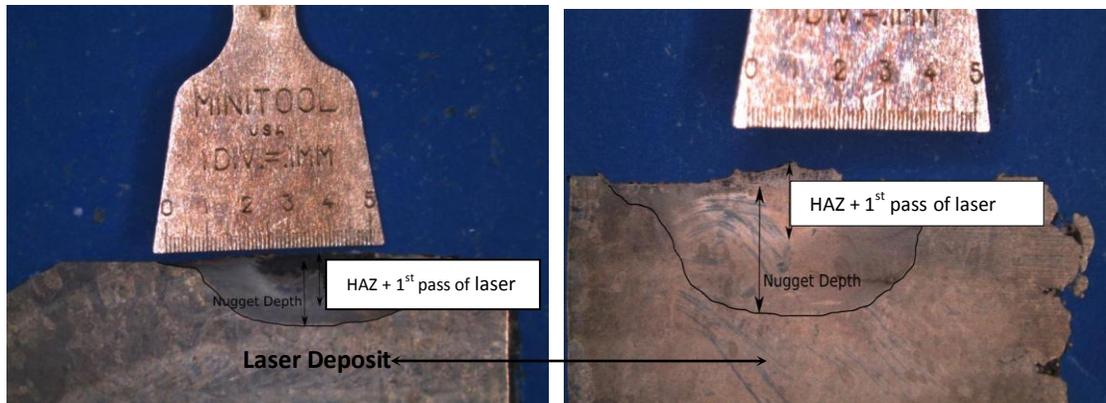


Figure 7. (a) Run no: T1-103007.2

(b) Run no: T2-102907.2

Table 4 Single laser scan

Run No:	Avg FSP Nugget depth (μ)	1 st Pass laser deposit + HAZ (μ)	Unaffected SZ (μ)
T1-103007.2	1600 \pm 62	1300	400 \pm 30
T2-102907.2	2700 \pm 32	1550	1350 \pm 34



Figure 8. (a) Run No: T1-103007.2

(b) Run no: T2-102907.2

Table 5 Double laser scan

Run No:	Avg FSP Nugget depth (μ)	D1(μ)	D2(μ)	Unaffected SZ (μ)
T1-103007.2	1600 \pm 17	500	1300	500 \pm 16
T2-102907.2	2550 \pm 35	850	1500	1300 \pm 29

D1=2nd laser pass deposit height + HAZ; D2=HAZ from 1st laser pass

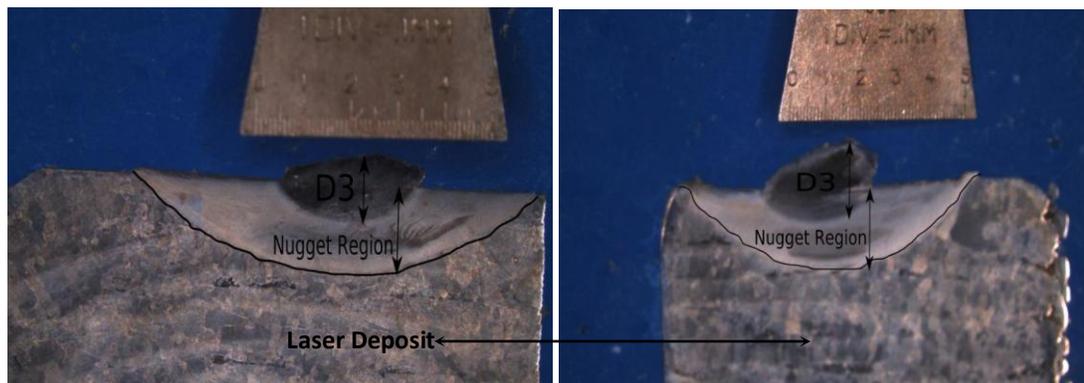


Figure 9. (a) Run no: T1-103007.2

(b) Run no: T2-102907.2

Table 6 Triple laser scan

Run No:	Avg FSP Nugget depth (μ)	D3(μ)	Unaffected SZ (μ)
T1-103007.2	1800 \pm 27	1300	800 \pm 24
T2-102907.2	2200 \pm 36	2200	1100 \pm 24

D3=3rd Pass laser deposit + HAZ (μ)

It can be seen that the nugget regions of the macro cross-sections shown in Figure 9 are parabolic and not flat towards the end of the SZ as has been observed in Figures 7 and 8. The cause for this is directly dependent on the temperatures existing at the time of processing this particular section of the run. Although the laser interacted thrice in this region it is not expected to have caused a parabolic nugget because a well defined HAZ from the third pass of the laser beam was noticed. For the stirs performed with Tool-2, the unaffected region in the stir zone which was comprised of the equiaxed α grains [6] ranged from 1100 μ m-1300 μ m and for Tool-1 it ranged from 372 μ m-834 μ m. It became clear from the analysis that the SZ nugget would not be completely eliminated and that around 50-60% of the original stir depth will be unaffected after the subsequent laser deposition. Tool-2 provided more unaffected volume and hence it would be an ideal choice for the FSP wall building process.

4.2. BUILD METHODOLOGY

The proposed build methodology is applicable to building a layered, fully forged, recrystallized, wall like structure with Ti-6Al-4V as the material of construction. The deposition parameters required for this build process have been mentioned in Table 7.

Table 7 Deposition parameters

Laser Power(kW)	Laser Scan Speed	Powder Feed Rate
1	6.35 mm/s (15 IPM)	1.5 gms/min

The desired build height or geometry can be achieved in 4 major steps and each of these steps would consist of a series of operations utilizing the full hybrid laser deposition capability of the LAMP system.

4.2.1. Step-1: Substrate welding

- (A) Deposition of 1st Macro layer consisting of 4 laser scans
- (B) Preparing the flat surface for FSP by milling ~1mm (0.04 in)
- (C) Friction Stir Processing I
- (D) Friction Stir Processing II with 50 % overlap with the first pass.
- (E) Final machining for removal of the flash generated from FSP I and FSP II, ~ 0.25 mm (0.01 in) material removal.

1st macro layer will be made up of 4 layers of laser scans. It was observed during experiments that 4 layers would yield approximately 1.75mm of deposit and this would ensure tool contact with the substrate. A detailed schematic of the step-1 process has been shown in Figure 10.

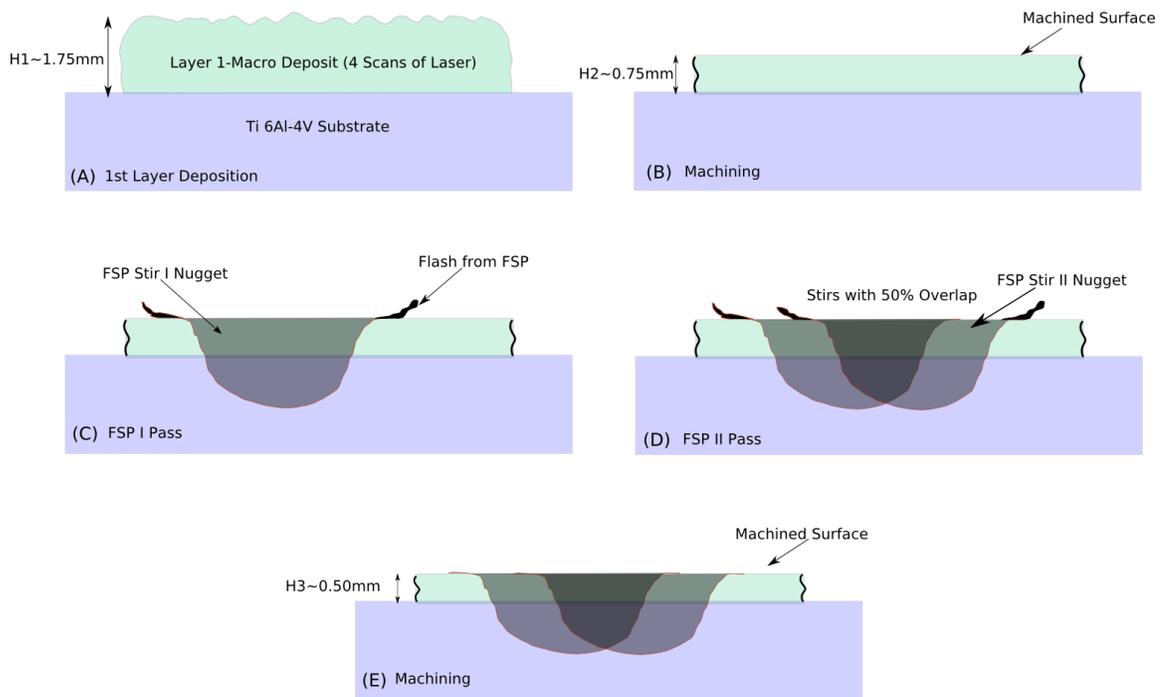


Figure 10. Step-1 Substrate welding with 50% overlap

1 mm of the deposit would have to be milled off to leave the flat surface for FSP. This would be followed by FSP I and FSP II which would be performed with 50% overlap in order to obtain maximum volume of the forged microstructure. Final machining will be done to prepare the structure for the next layer of deposition.

4.2.2 Step-2: Deposition over friction stirred deposit

- (A) Deposition of Macro layer-2 consisting of 6 laser scans
- (B) Preparing the flat surface for FSP by milling ~1mm(0.04 in)
- (C) Friction Stir Processing III
- (D) Friction Stir Processing IV with 50 % overlap with the FSP III pass.

(E) Final machining for removal of the flash generated from FSP III and FSP IV, ~
0.25 mm (0.01 in)

The 2nd macro layer will consist of 6 laser scans. This is different from the earlier step because penetration of the tool into the substrate had to be ensured in step-1 which is not the case in this step. It has to be only ensured that the tool is able to stir through the HAZ created due to the subsequent deposition over the stir zone in this particular step and all the subsequent steps. The schematic for step-2 has been shown in Figure 11.

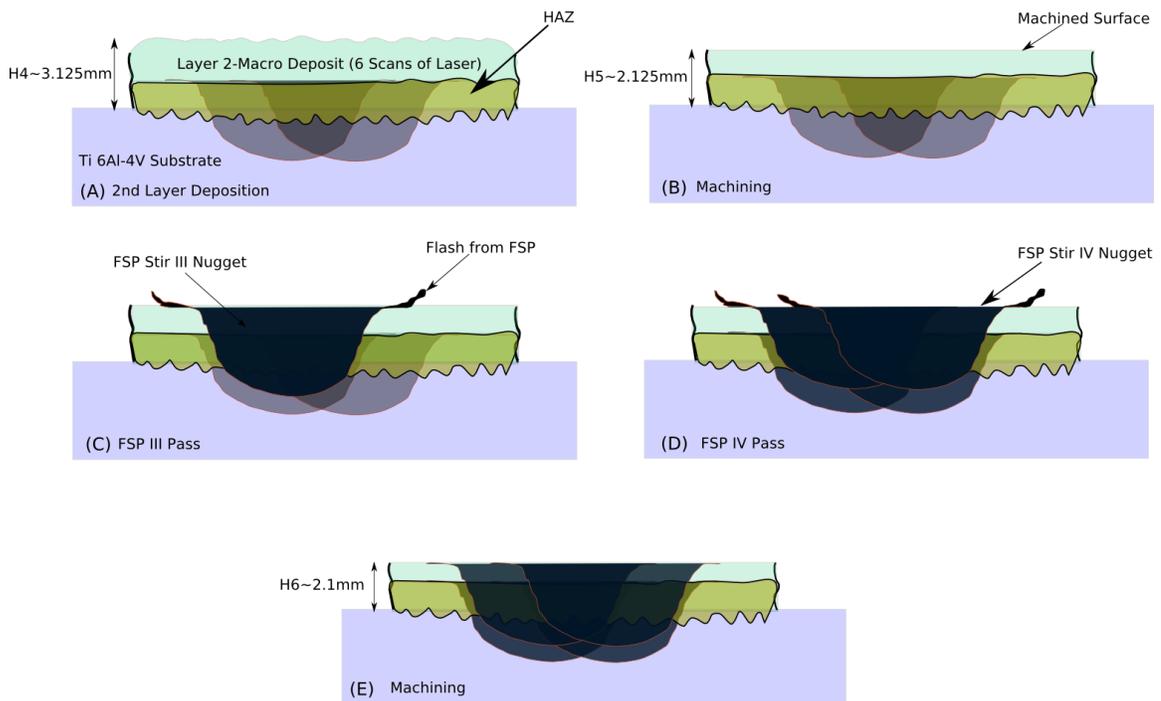


Figure 11. Step-2 Subsequent laser deposition

4.2.3. Step-3: Deposition of subsequent steps (N-1) x (Step-2). This would be a repetition of the Step-2 process (N-1) times, where N represents the total number of

layers that will be deposited over Step-1. Figure 12 shows the schematic of the deposited structure after the completion of Step-3. At this stage the desired height of the structure would have been attained.

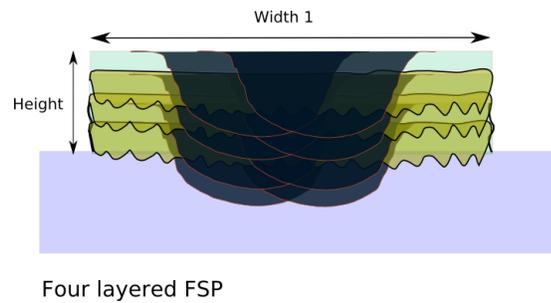
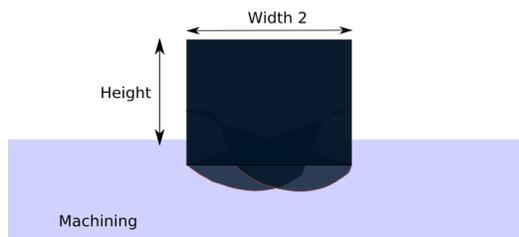


Figure 12. Step-3 Completed laser deposition

4.2.4. Step-4: Final machining

The final step in building the structure with fully recrystallized microstructure would be to modify the intermediate structure obtained after Step-3 and machining off the sides of the deposit to leave behind a fully forged structure. The schematic of the obtained structure is shown in Figure 13.



Fully Recrystallized Ti-6Al-4V Structure after Machining

Figure 13. Final structure

5. CONCLUSIONS AND FUTURE WORK

A build methodology for developing a fully forged microstructure by integrating two innovative and relatively new technologies of friction stir processing and hybrid laser deposition has been proposed. Friction stir processing parameters which will result in stirs with better surface quality and nugget integrity for laser deposited Ti-6Al-4V has been identified. It was proved that subsequent laser deposition over the stir zone does not completely change the microstructure of the stir nugget. Thus components prepared using this technique will consist of highly refined microstructure throughout its build volume. The components prepared with this technique can be of great importance in aerospace industry. More research will be required in order to optimize the volume of the nugget region unaffected by subsequent deposition. Experiments are in progress to demonstrate the proposed methodology and initial tests have shown encouraging results.

6. ACKNOWLEDGMENT

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VITA

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