



Prevention of Liquation Cracking In Alloy 718 Laser Depositions Using Friction Stir Surface Processing

Prepared by:
Levi Lange

Faculty Advisors:
Dr. Michael West
REU Program Director

Dr. Christian Widener
AMP Center Director

Dr. Bharat Jasthi
AMP Center Research Scientist

Dr. Alfred R. Boysen
Professor, Department of Humanities

Program Information:
National Science Foundation
Grant # 1157074

Research Experience for Undergraduates
Summer 2013

South Dakota School of Mines and Technology
501 E Saint Joseph St.
Rapid City, South Dakota

Table of Contents

Abstract	3
Introduction	4
Broader Impact	5
Procedure	6
Base Material Analysis	6
Material Processing	10
Results	11
Processing	11
Microstructural	14
Mechanical	21
Discussion	24
Conclusion	24
Summary	24
Future Work	25
References	26
Acknowledgments	27

Abstract

Inconel 718[®], a new γ'' strengthened, nickel-based superalloy, is intended for use in high temperature applications related to both aerospace and industrial gas turbines. These components are suffering from simple wear and tear during use. Therefore Industry is faced with the problem of trying to repair these parts rather than replace them due to cost. Friction Stir Processing (FSP), a new materials processing technique invented by The Welding Institute, along with laser deposition to attempt to repair these components. This study attempted to evaluate the feasibility and results of Friction Stir Processing 718 using an MTS ISTIR-10 Intelligent Friction Stir Welder, and the Optimec Laser Engineered Net Shaping (LENS) System. The welds were then sectioned, heat treated, and used for metallography and microhardness samples.

It was found that FSP paired with Laser Deposition is indeed possible, though further characterization needs to be addressed to determine if it is a feasible repair technique. Also, FSP refines the microstructure of 718 alloy and increases its microhardness. Lastly, the heat treatment allowed for the hardness of the deposition to be recovered.

Introduction

Inconel 718 alloy is a recently developed precipitation hardened; nickel-based superalloy designed to exhibit exceptional corrosion resistance, strength, and toughness up to 650° C. This alloy is most often used in cast form in high temperature applications such as aerospace or industrial gas turbine components. Being that these components are in the as cast form, they naturally have very large grains, which in turn creates large grain boundaries causing more grain-boundary segregation, and chances for liquation cracking. Dr. Jasthi, Dr. Howard, and Dr. West have introduced the idea that Friction Stir Welding/Processing may be applied to nickel-based superalloys to refine their microstructures and reduce the grain sizes, potentially preventing liquation cracking.

Liquation cracking is intergranular hot cracking that occurs in the Partially Melted Zone (PMZ) of a weld, resulting from localized melting at grain or other boundaries, combined with the thermal strains associated with welding (4). It refers to the total liquid present in the interdendritic regions as well as the grain boundaries. Liquation cracking can occur during fabrication by welding in either the heat affected zone in the parent material, or in previously deposited weld metal during a subsequent run (4).

The goal of this preliminary study is to develop a means to repair these cast turbine components after they have failed in service. Normal means of welding do not work and because of the cost of the material replacing these components is also not an option. Therefore we will need to repair them back to where they meet required specifications to be deemed safe for re-use.

The FSP has already been run on the properly heat treated 718 alloy samples. So what needs to be done to accomplish these goals is to take samples of 718 alloy that have been friction stir processed, and samples that are strictly parent material, no secondary processing, and laser

depositing a weld of 718 alloy onto these samples, and run it at varying wattages. After the laser deposition, some samples, both parent and FSP, will be saved to go through a secondary heat treatment. All samples will be polished and etched in order to examine the microstructure, and obtain some representative data of the effects of FSP, laser deposition at varying wattages, and secondary heat treatments.

Broader Impact

As mentioned earlier, Friction Stir Welding and Processing is an advanced material-joining technique that is beginning to be used widely in industry. Friction Stir Welding and Processing has many advantages including refining the base metal microstructure, ensuring homogeneous distribution of precipitates, and possibly eliminating detrimental phases. If these advantages can be utilized on cast components of Inconel 718 superalloys, it could save the aerospace and industrial turbine industry a lot of time, money, and resources attempting to replace or fix these parts. It would give them a relatively fast and cost effective means of repairing these very expensive pieces of machinery.

Examining the way the microstructure of the cast 718 alloy is affected by the friction stir processing could lead to discovering additional fabrication techniques, or treatments for Inconel 718. This would in turn increase the versatility and usefulness of the high strength alloy, which could prove very useful in other industrial applications.

Procedure

Base Material Analysis

An Inconel 718 alloy cast plate in final heat treated condition was obtained from a former student. This plate was friction stir processed by this previous student using a Tungsten (W) – Rhenium (25% Re) – Hafnium Carbide (4% HfC) pin tool, and the MTS ISTIR-10 Intelligent Friction Stir Welder, with a Megastir cooling head and argon shroud. The parameters that were used to develop consistent welds were a rotational speed of 200 RPM, a traveling speed of 2 IPM, and a downward force of 5,000 lb forge force. A schematic of the pin tool can be seen in Figure 1. The 718 alloy plate with the friction stir processing can be seen in Figure 2 following, and Table 1 shows the alloying elements that make up Alloy 718.

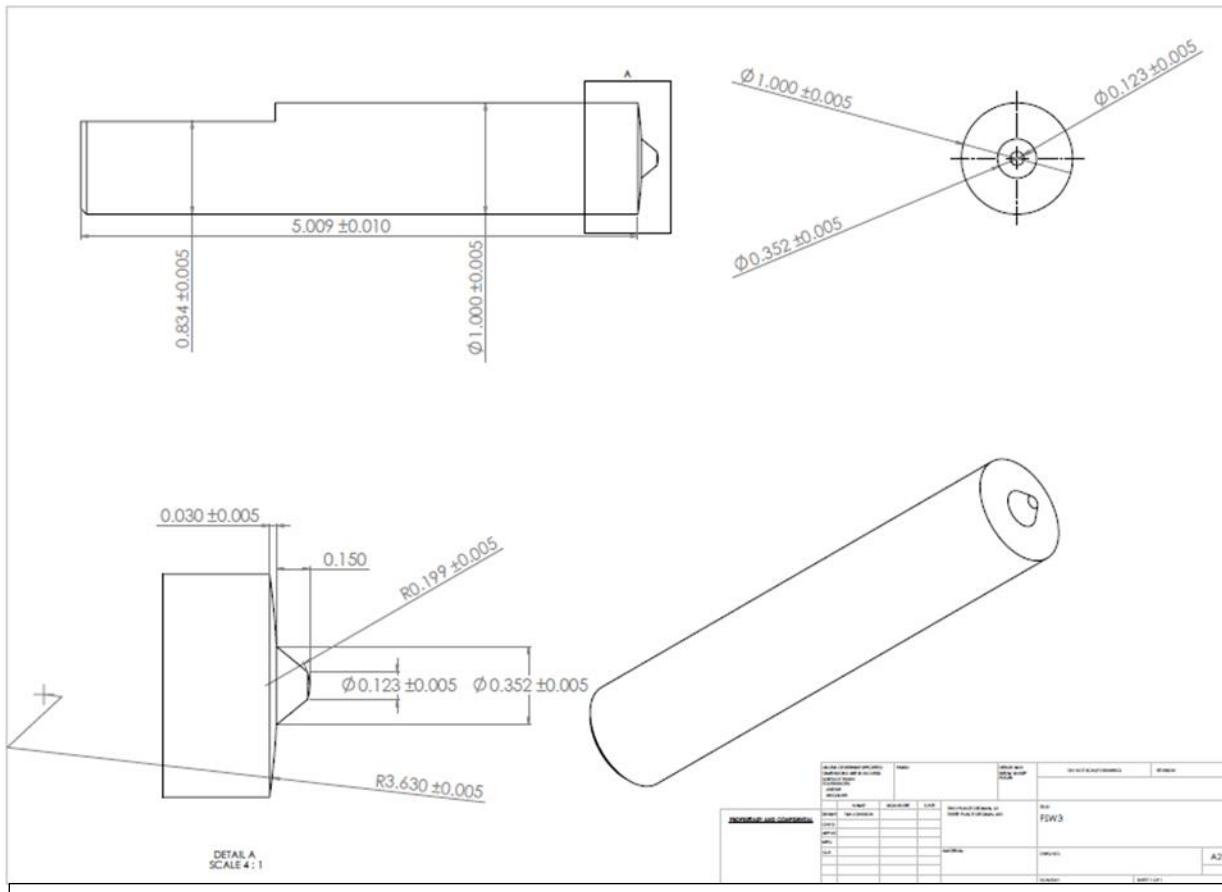


Figure 1. Tungsten-25% Rhenium-4% Hafnium Carbide Pin Tool Technical Drawing. (Photo Courtesy of Todd Curtis)

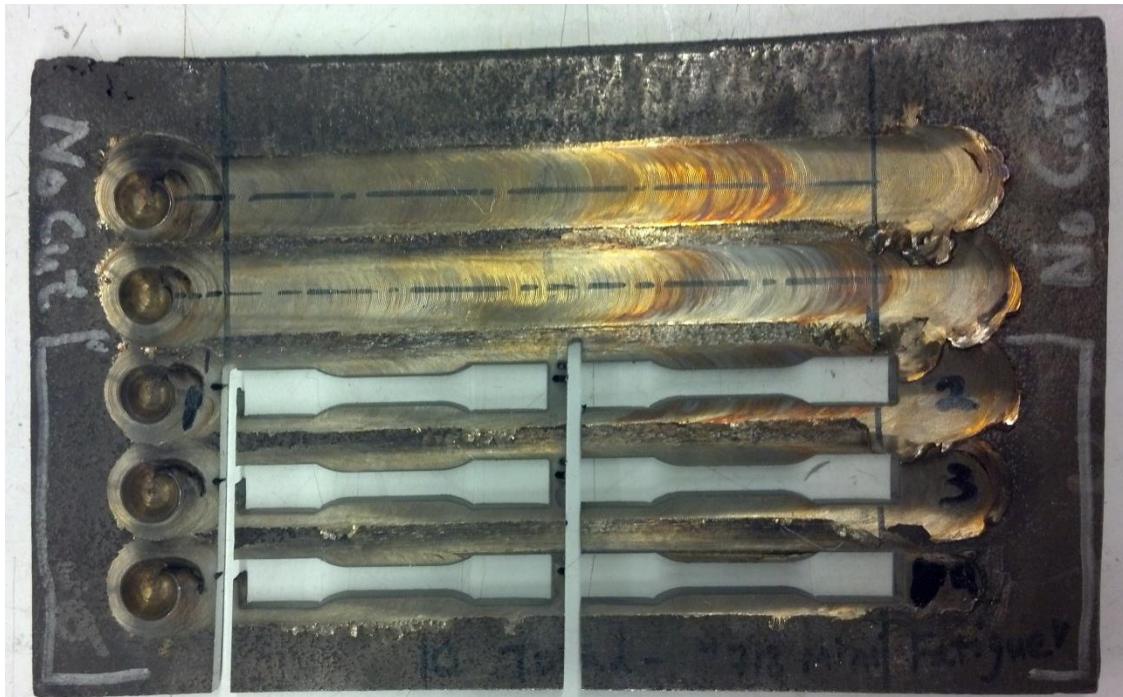


Figure 2. 718 Alloy Plate with Friction Stir Processing zones, and As-Cast zones (Author's Work)

Table 1. 718 Alloy Composition by weight percent (Courtesy of Dr. Bharat Jasthi.)

Alloying Element	Weight Percent (%)
Nickel (Ni)	55%
Chromium (Cr)	21%
Iron (Fe)	11.15%
Niobium (Nb)	5.5%
Molybdenum (Mo)	3.3%
Aluminium (Al)	1.15%
Columbium (Cb)	1%
Copper (Cu)	.8%
Mn, Ti, Si, C, S, P, B	< .5%

Because the material was already in the final heat treated condition and the friction stir processing was already completed, samples were able to be taken from both the friction stir processed zones as well as the cast material which will be referred to now as the “parent” material. One sample from each zone was cut out of the plate using the Maxiem 1530 jet cutting

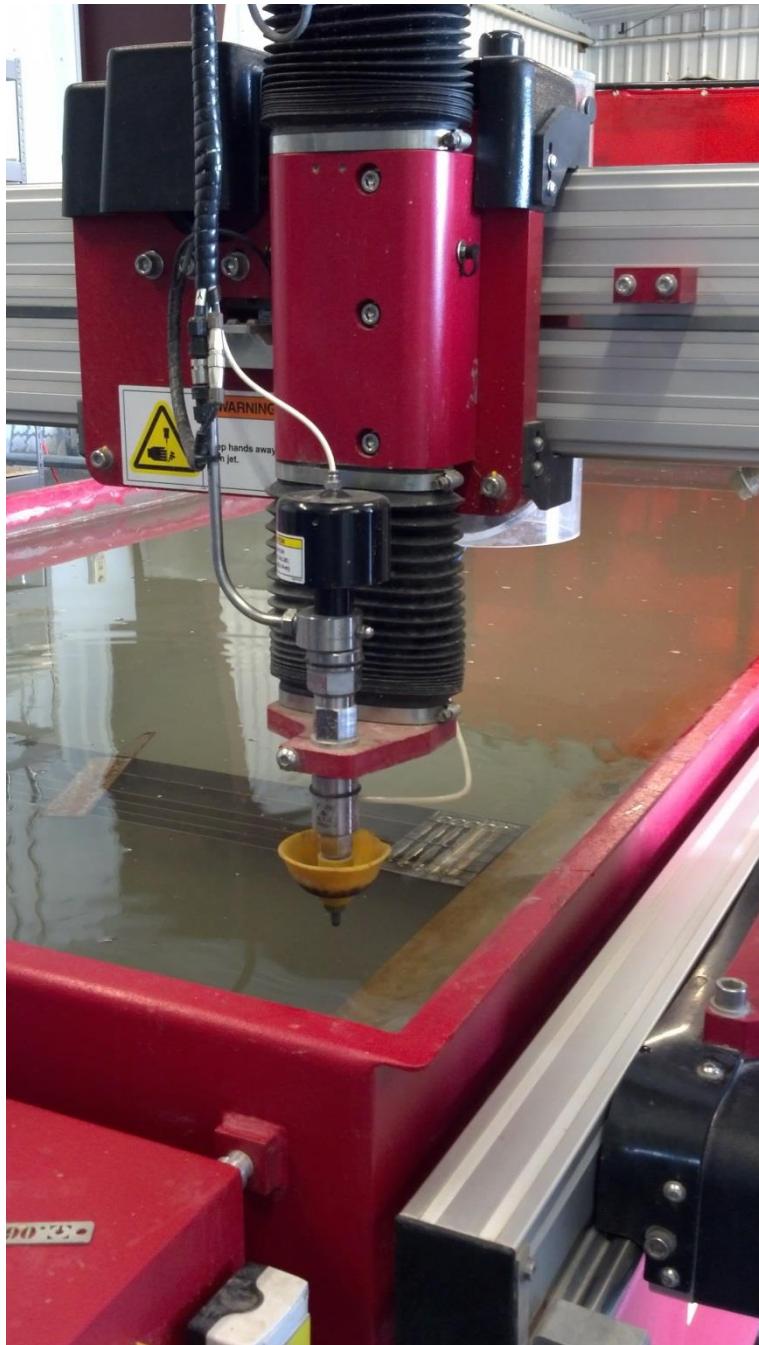


Figure 3. Maxiem 1530 Jet Cutting System, Cutting courtesy of AMP. (Author's Work)

center water jet system. This system with the plate fixed into place can be seen in Figure 3 above.

The samples that were cut were then hot mounted using the LECO® Black Bakelite Powder, and grinding using successively finer grits of silicon-carbide paper (80, 240, 400, 600, 800, 1200 grit). Polishing was then performed using successively finer polishing pads with its corresponding LECO Ultra Diamond Suspension (9, 6, 3, 1, 0.5 micron), with varying time and RPM, that gave the proper results.

The polished samples were then etched by a wiping motion, using a cotton swab, and Walker's Etchant. The chemical makeup of Walker's Etchant can be seen in Table 2 below.

Table 2. Walker's Etchant chemical makeup. (Courtesy of Dr. Bharat Jasthi.)

Walkers Etch	
Volume (ml)	Chemical
50 ml	Hydrochloric Acid (HCl)
10 ml	Phosphoric Acid (H_2PO_3)
10 ml	Hydrofluoric Acid (HF)
30 ml	Nitric Acid (HNO_3)
50 ml	Distilled Water (H_2O)
20 ml	Acetic Acid ($CHCO_2H$)
15 g	Iron Chloride ($FeCl_3$)

The samples were then rinsed with water following etching and dried with compressed air. The finished samples were observed using an optical microscope for microstructure, and grain size. Finally Vickers microhardness measurements were taken. The same preparation and testing procedures that were performed on the parent and friction stir processed materials were repeated on the following samples as noted below.

Material Processing

Once the parent material and the friction stir processed materials had been characterized, six more samples were cut using the water-jet cutting system, three from the parent material, and three from the friction stir processed material. The samples were then individually placed into the Optimec Laser Engineered Net Shaping (LENS) System. The system laser deposited an Inconel 718 powder, developed by Starmet Powders, with a mesh size of -140 +325 (102-45 micron) that ran with an argon carrier gas at 2 L/min, an argon purge gas at 30 L/min, and a powder feed of 15 g/min. The laser deposited 30% overlapping beads, which had a diameter of .066". All parameters that have been stated thus far for the laser deposition were fixed. The only varying parameter was the laser power. The power was varied from 500W to 700 W and finally to 900W. The exact buildup of the beads can be seen in the macrographs in Figure 4 respectively.

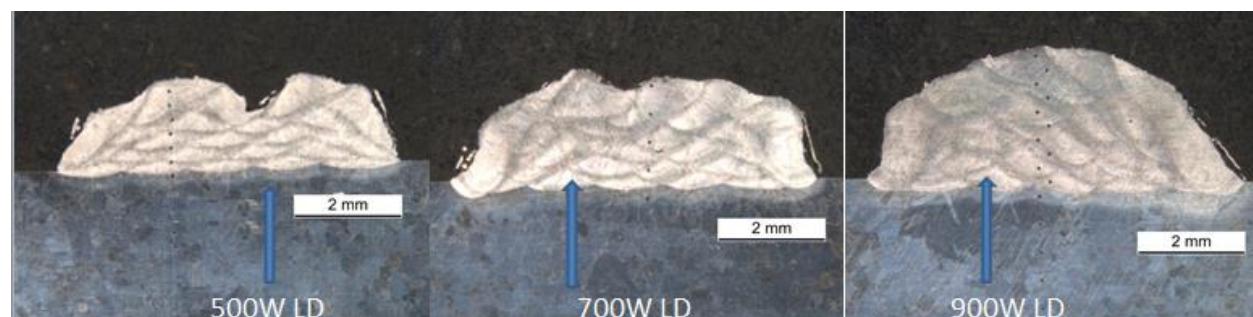


Figure 4. Macrographs of the 718 alloy Laser Deposition buildup on 718 alloy base. (Author's Work)

After deposition, the six samples were then cut into half perpendicular to the laser deposition direction using a wet saw. Half of the samples, three from the parent and three from the friction stir processed, were run through a post weld heat treatment. The AMS5383E, section 3.5 Specification post weld heat treatment is a three step treatment: 1) Homogenization: Heat to $2000^{\circ}\text{F} \pm 25$ ($1093^{\circ}\text{C} \pm 14$), hold at heat for 2 hours and cool at a rate equivalent to an air cool or faster to 900°F (482°C). 2) Solutionizing: Heat in a suitable protective atmosphere to a temperature within the range of 1750 to 1800°F (954 to 982°C), hold at the selected temperature for no less than one hour, and cool at a rate equivalent to air cool or faster. 3) Precipitation Hardening: Heat to $1325^{\circ}\text{F} \pm 15$ (718°C), hold at heat for 8 hours, furnace cool to $1150^{\circ}\text{F} \pm 15$ (621°C) at an average rate of $100^{\circ}\text{F} \pm 15$ (55°C) per hour, hold at $1150^{\circ}\text{F} \pm 15$ (621°C) for 8 hours and cool at a rate equivalent to air cool, for a total of 22.75 hours.

After the post weld heat treatment all twelve samples, six with the post weld heat treatment, and six in the as deposited form were all mounted in the LECO® Black Bakelite Powder, sanded, polished, and examined for microstructure and microhardness in the same manner as the initial parent no laser deposition and friction stir processed no laser deposition samples were, as stated in Base Material Analysis.

Results

Processing

The power of the laser had a large impact on the quality of the buildup of the weld. The quality and build height increased as the wattage of the laser increased. This can be seen in the macrographs of the laser depositions above in Figure 4. The power of the laser also seemed to have a small influence on the severity of the liquation cracking on the parent materials. Below is

a graph showing the maximum length of cracking on the three different powers. It can be seen that the increasing power led to an increase in maximum crack length. Another aspect of the processing that had an effect on liquation was the post weld heat treatment. The cracking in the cast material with the post weld heat treatment showed a more severe cracking zone and heat affected zone. Figure 6 below shows a liquation crack in a 700W parent material deposition in the pre-heat treated condition and in the post-heat treated condition.

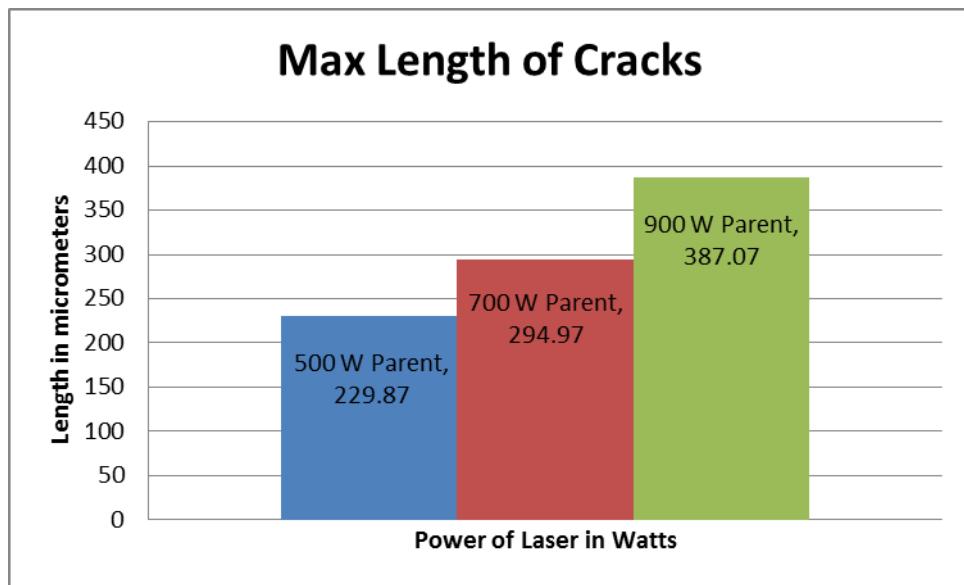


Figure 5. Graph of the Average crack length corresponding to laser power. (Author's Work)

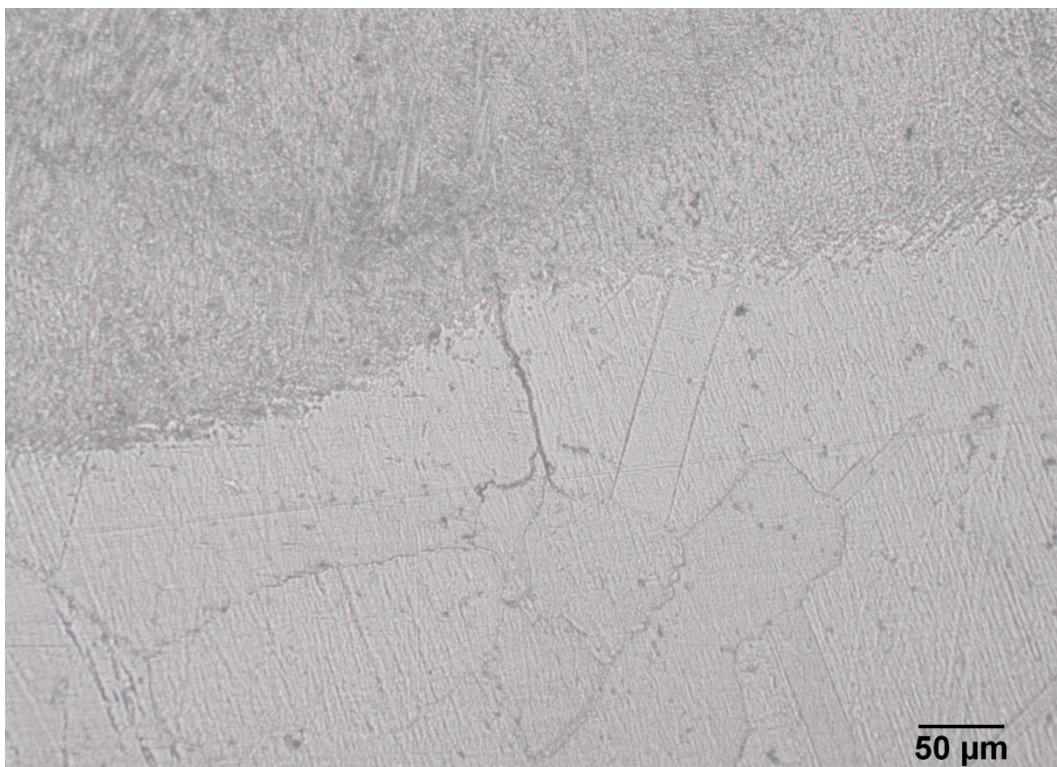


Figure 6. Optical Microscopy of liquation cracking in 700W LD Parent Material before heat treatment above, and post heat treatment below. (Author's work)



Microstructural

The initial metallography from the parent material and the friction stir processed material showed that there was a large reduction in grain size from approximately $276 \mu\text{m}$ to $< 5 \mu\text{m}$. Figure 7 and Figure 8 shows a macrograph of the decrease in grain size from the parent material to the friction stir processed material respectively.

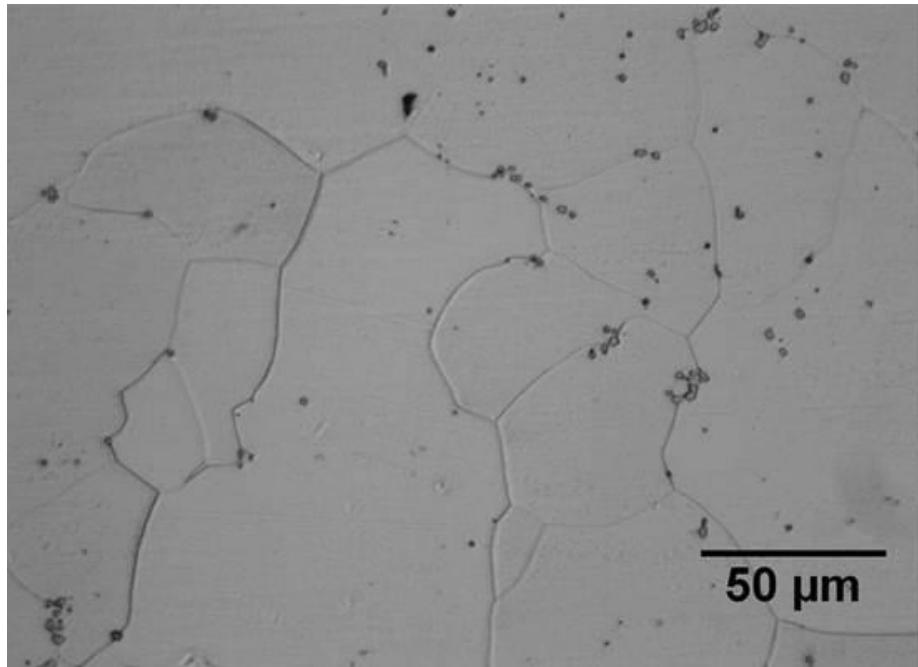


Figure 7. Micrograph of the as cast 718 alloy (Author's Work)

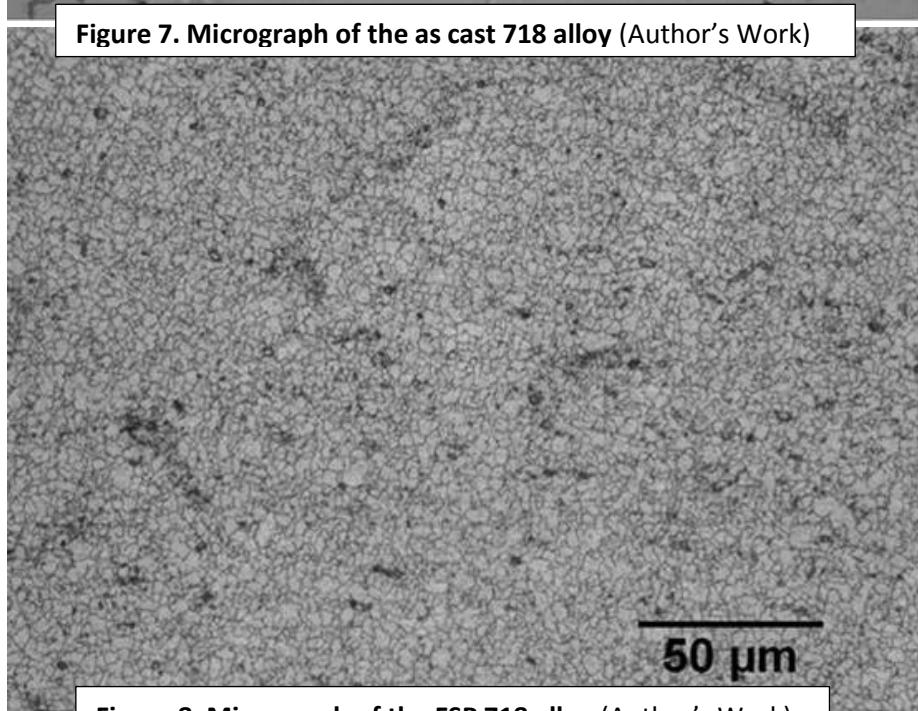


Figure 8. Micrograph of the FSP 718 alloy (Author's Work)

The next area that was examined was the parent material samples with the varying laser powers. As said earlier the increasing laser power caused an increase in maximum crack length. The following Figures and table show some of the micrographs that were taken showing the parent material, the laser deposition, and the liquation cracking that occurs due to the laser deposition.

Following the parent materials microstructural examination was the friction stir processed material with the varying laser deposition powers. No liquation cracking was observed in any of the samples that were tested and examined under the optical microscope. This is the outcome that was ultimately the goal of the entire project. The friction stir processing decreased the grain size, decreasing the area for liquation cracking to occur. Figures 9, 10, 11 immediately following show some of the micrographs of the laser deposition on the friction stir processed material.

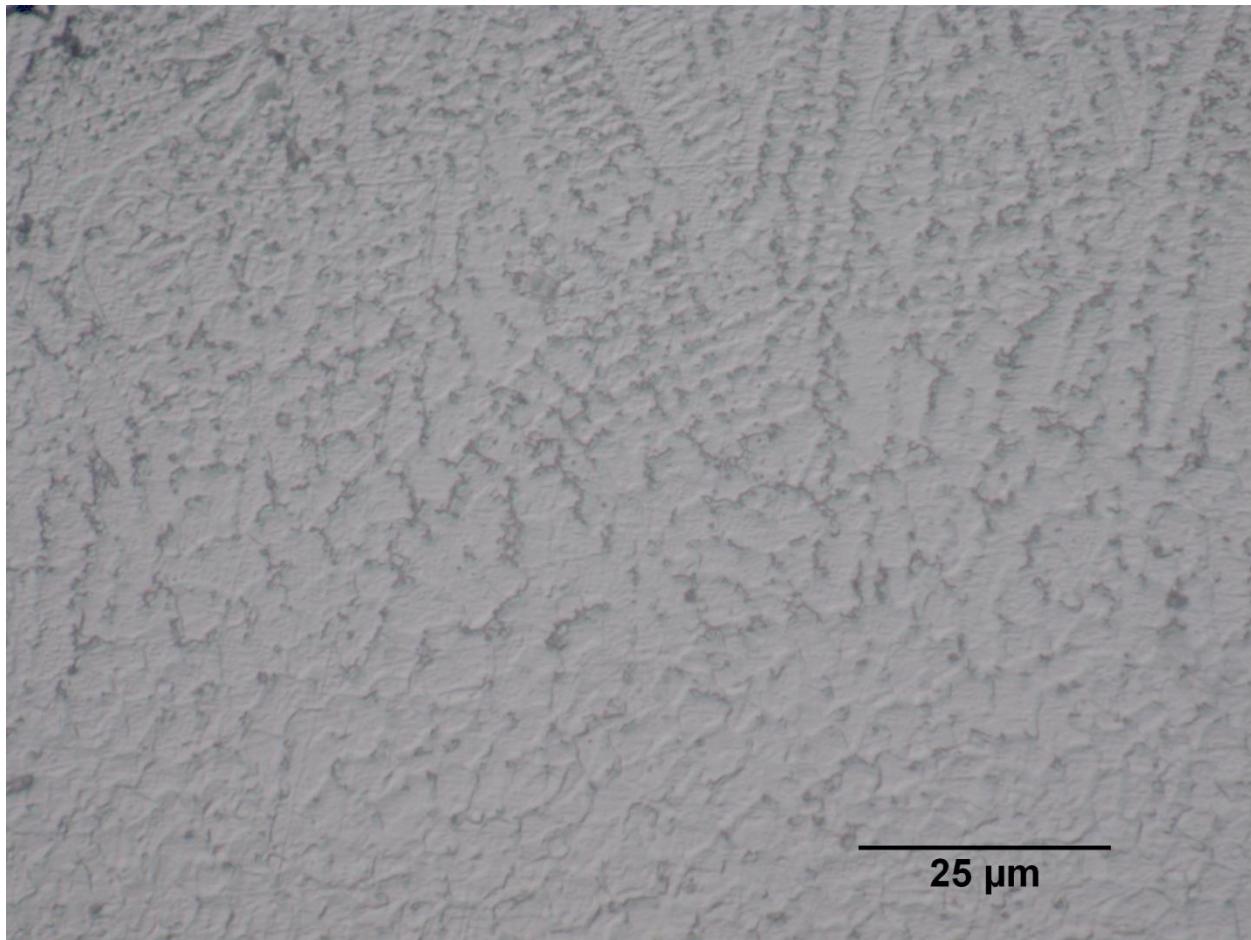


Figure 9. Micrograph of the FSP 718 alloy with 500 W Laser Deposition (Author's Work)



Figure 10. Micrograph of the FSP 718 alloy with 700 W Laser Deposition (Author's Work)

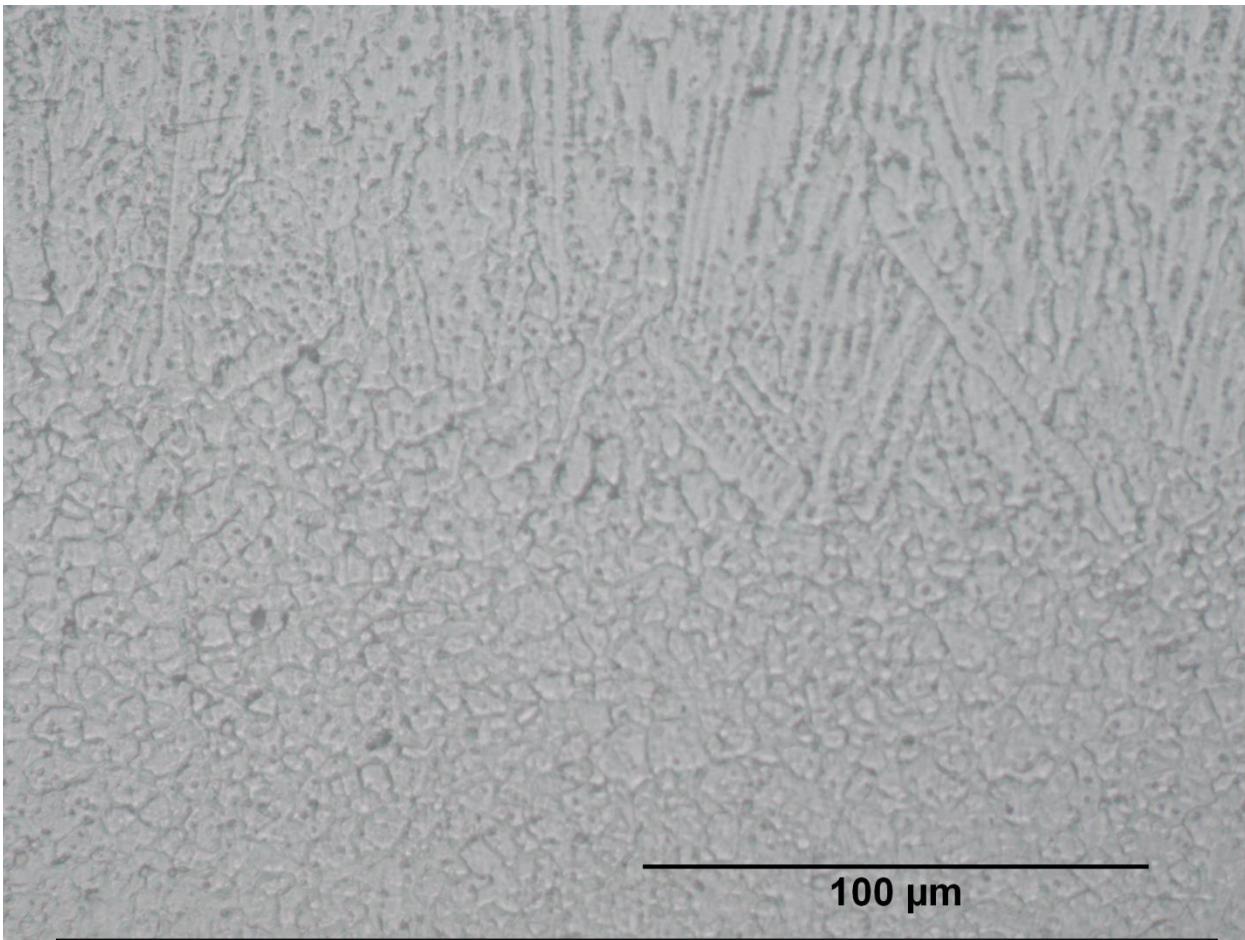


Figure 11. Micrograph of the FSP 718 alloy with 900 W Laser Deposition (Author's Work)

The same examination of the pre-weld heat treated parent and friction stir processed materials were performed on the post-weld heat treated parent and friction stir processed materials. As stated earlier, the liquation in the parent depositions after the heat treatment was greatly affected. The extent of liquation increased after the heat treatment in that the cracks were thicker, and had a large heat affected zone around many of the cracks. Some of the micrographs showing these features can be seen in Figures 12 and 13 following.

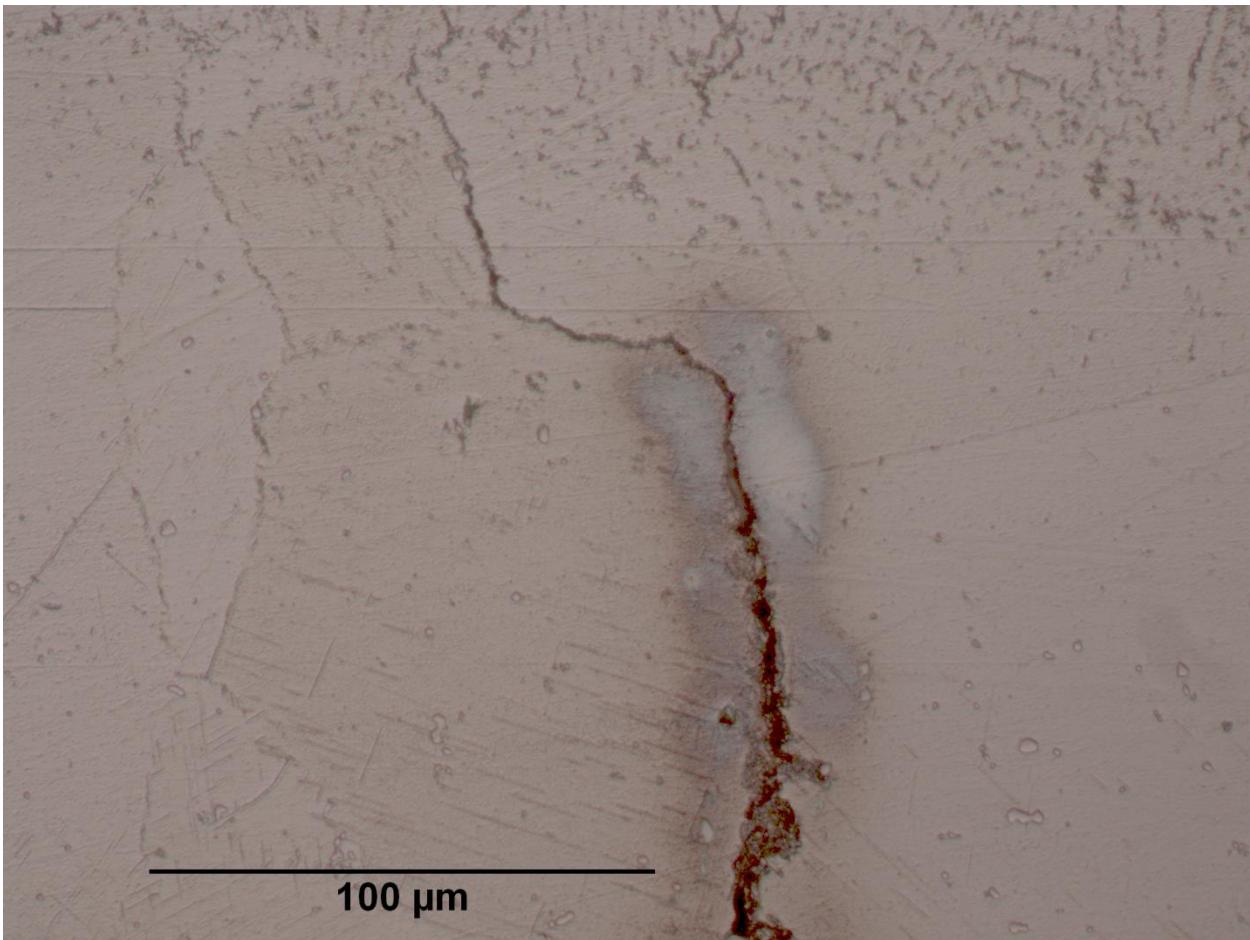


Figure 12. Micrograph of liquation cracking on 718 parent material with 700W Laser Deposition in Post-Weld Heat Treated condition. (Author's Work)

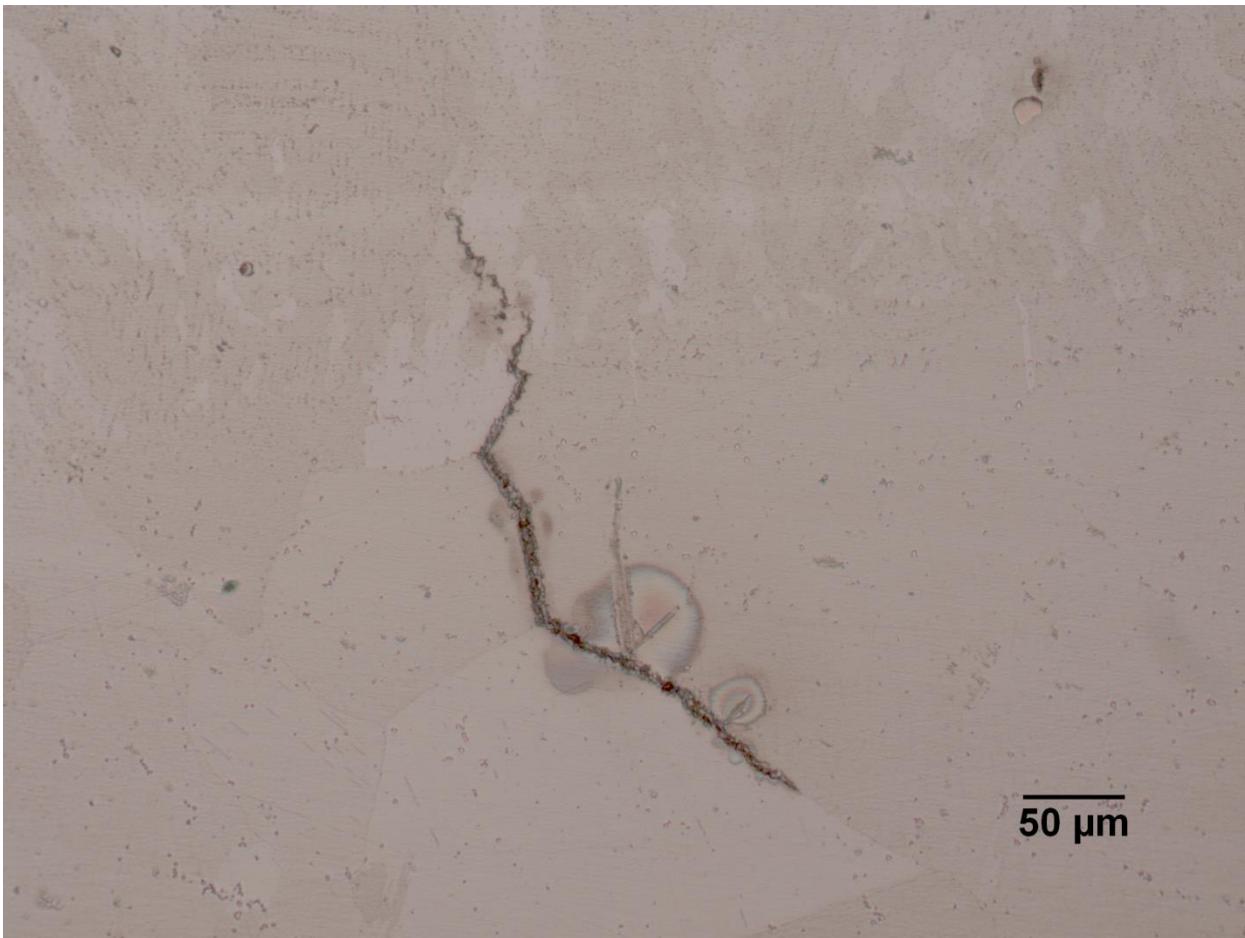


Figure 13. Micrograph of liquation cracking on 718 parent material with 900W Laser Deposition in post weld heat treated condition. (Author's Work)

Much like the pre-heat treated friction stir processed material with the laser depositions showing no liquation cracking, the post heat treated friction stir processed material with the laser depositions also showed absolutely no visible liquation cracking. These micrographs can also be seen in the Figures following. The small grain sizes made it difficult to see the grain boundaries very well under the optical microscope. This called for the need of the Scanning Electron Microscope (SEM). Unfortunately up to this point in time there has not been enough time in order to accomplish this. Following the microstructural analysis of the twelve samples, was the mechanical testing.

Mechanical

As a further test to characterize the starting condition of both the as cast 718 alloy and the friction stir processed 718 alloy, Vickers microhardness measurements were performed. The average hardness value of the parent material was found to be 437 HV. This shows that the material was in the final heat treated condition, being precipitation hardened. The average hardness value of the friction stir processed material was also taken and found to be 359 HV, showing that the material after the friction stir processing was still very close to that of the parent material. These measurements also provided a baseline for further microhardness measurements.

Microhardness measurements were taken for the parent material with the varying laser depositions, and were compiled into the graph in Figure 14. The laser deposition showed to be very soft, and the parent material even after the laser deposition still maintained its strength.

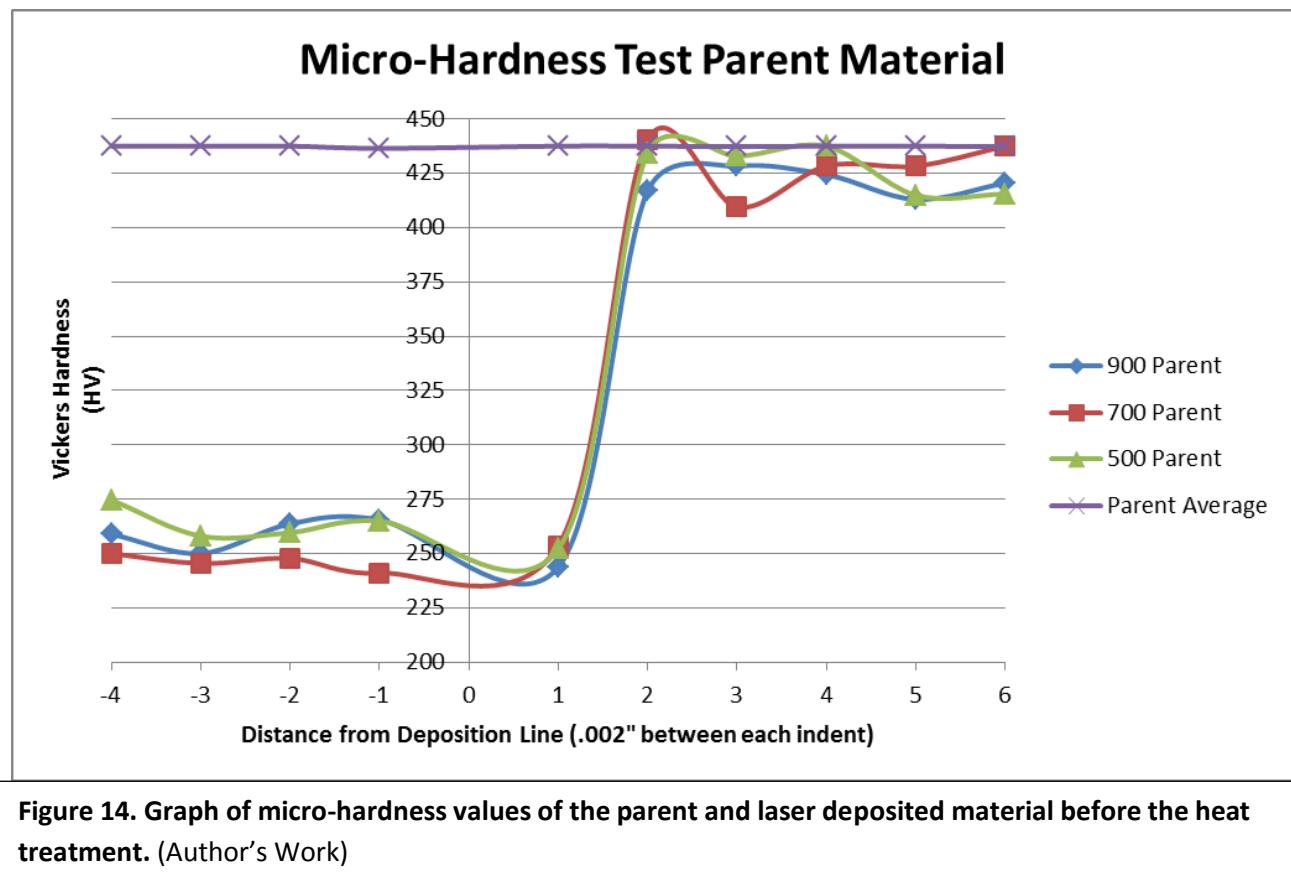


Figure 14. Graph of micro-hardness values of the parent and laser deposited material before the heat treatment. (Author's Work)

Microhardness measurements were then taken for the friction stir processed material with the varying laser depositions, and were compiled into the graph in Figure 15. The laser depositions again showed to be quite soft, and the graph showed a similar trend to that of the parent material with depositions. The friction stir processed area showed an increase from that of the deposition but not as much of an increase as the parent material.

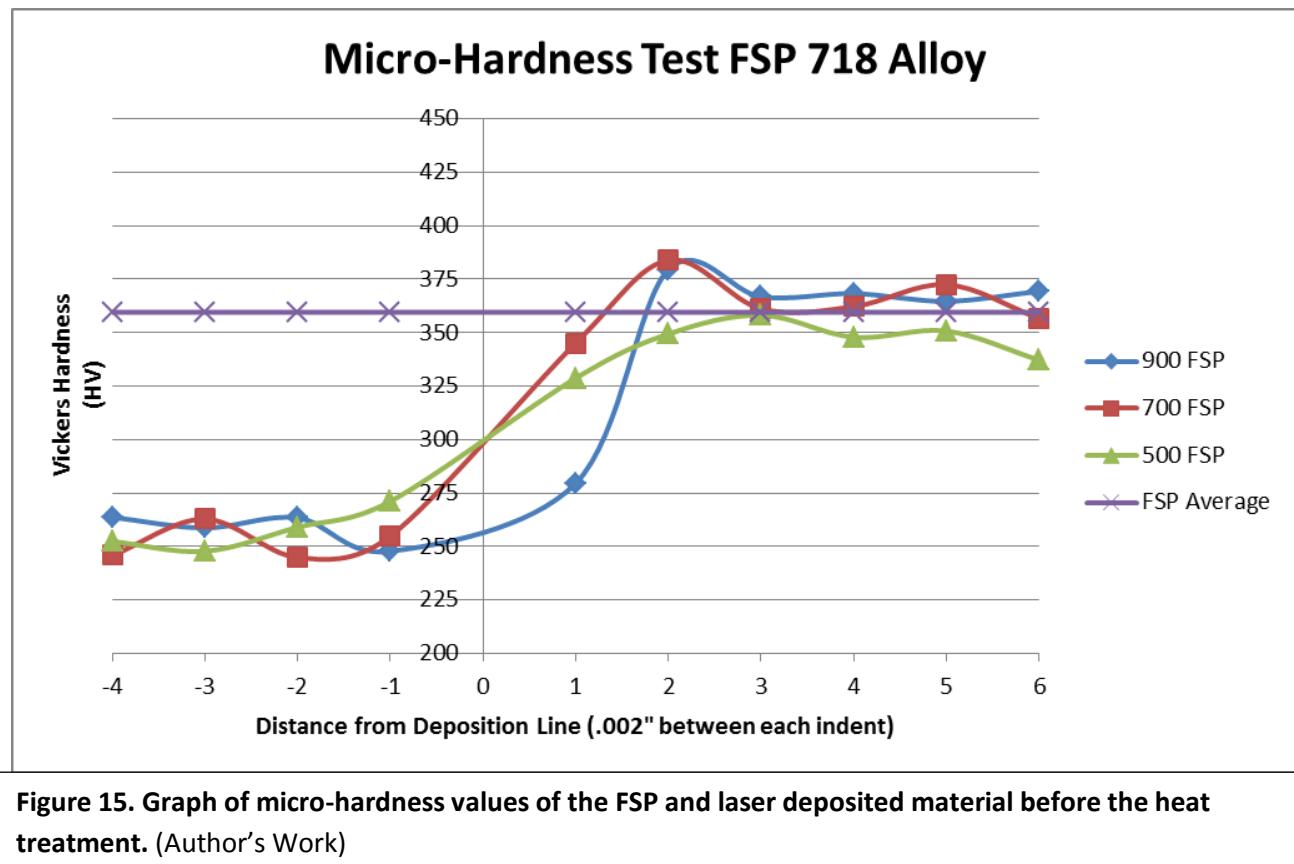


Figure 15. Graph of micro-hardness values of the FSP and laser deposited material before the heat treatment. (Author's Work)

Following was the microhardness characterization of the post-weld heat treated materials. The same procedure was followed as before, except an average was taken for each of the post-weld heat treated parent materials, and only one average was taken for the friction stir processed post-weld heat treated samples. The graphs following in Figure 16 and 17 shows that the heat treatment greatly increased the hardness of the laser deposited material, while maintaining hardness for the most part in the friction stir processed material. Further characterization of the

results for the parent material needs to be done due to unusual results.

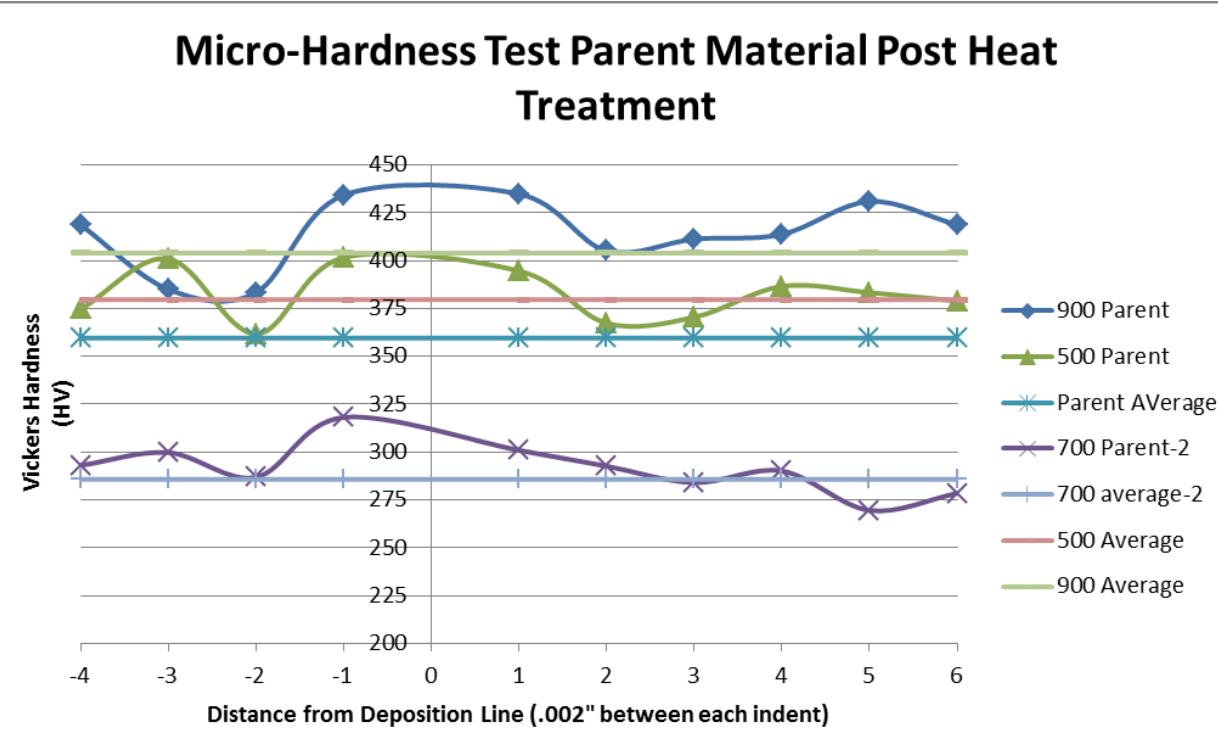


Figure 16. Graph of micro-hardness values of the Parent and laser deposited material after the heat treatment. (Author's Work)

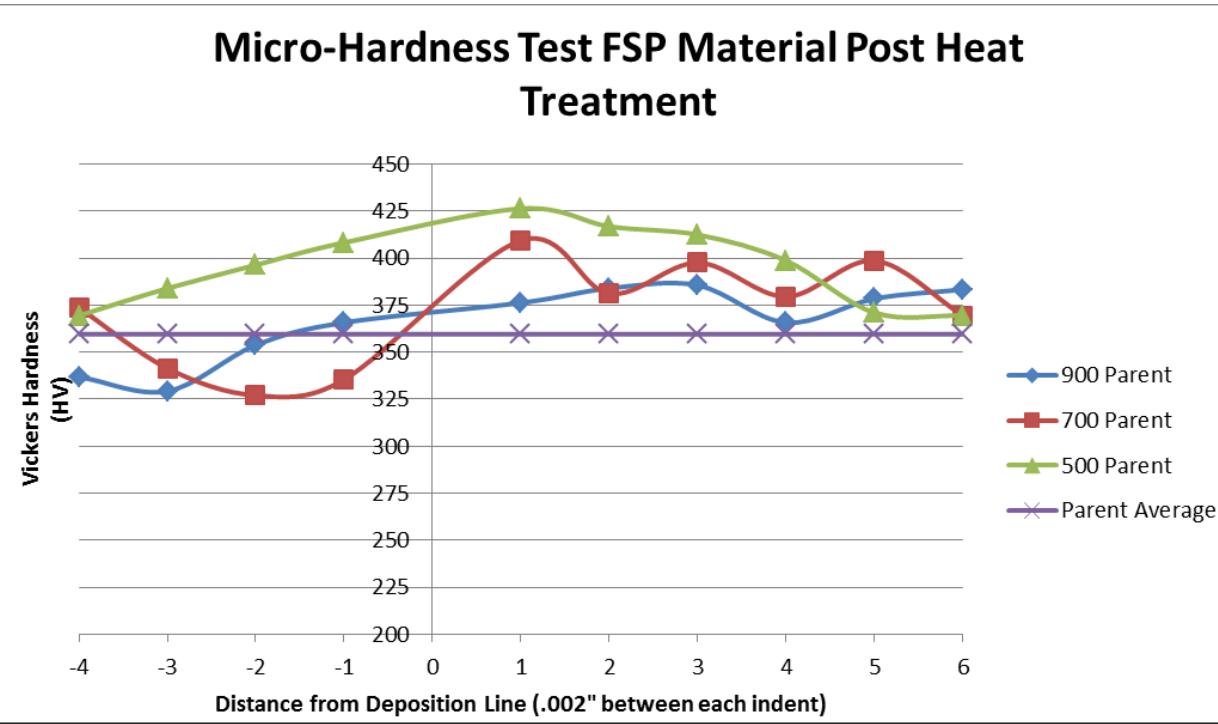


Figure 17. Graph of micro-hardness values of the FSP and laser deposited material after the heat treatment. (Author's Work)

Discussion

The liquation cracking seems to be completely prevented due to the friction stir processing. The optical microscope doesn't show any visible liquation but SEM characterization needs to be done to confirm this. The grain size refinement due to the friction stir processing is certainly the leading cause of liquation prevention.

The microhardness measurements in the laser depositions after the heat treatment were much higher than those before the heat treatment, this is a result of γ'' precipitation hardening. This shows that it could be possible to laser weld, then heat treat, and be able to maintain its mechanical properties. The friction stir processing also showed little to no detrimental effects on the microhardness, therefore making it even more of a viable option.

As a final note the heat treatment solved the problem of the soft laser deposition, but on the parent material, it caused the liquation cracking to become even more severe than before the treatment. Therefore, in order for the heat treatment to be beneficial it needs to be paired with the friction stir processing.

Conclusion

Summary

Much work remains to be done in order to really know if friction stir processing, laser depositing, and heat treating will really be a viable means to repair these 718 alloy turbine components, but the initial results from the testing show these steps to be very promising. The benefits of friction stir processing far outweigh the disadvantages. The parameters of the friction stir processing as well as the parameters of the laser deposition could be researched and adjusted to complement each other and allow for increased mechanical and microstructural properties all around, making it perfect for repair in the industrial and aerospace gas turbine industry.

Future Work

As was just said the friction stir processing and laser deposition parameters need to be worked together in order to produce the most desirable outcome of a defect free weld.

In order to get a better mechanical properties understanding, many more tests need to be run. These tests include tensile testing, fracture toughness, impact testing, etc. These will provide more clarity as to whether or not these steps to repair the components will meet the in-service needs. For example, if a defect free repair is accomplished, but the part is too brittle for the application, then it will not be applicable, and something will need to be adjusted.

Along with mechanical testing is more specific material testing, such as corrosion or creep testing. This information would be pertinent considering the component's intended usage in very hot and corrosive conditions.

References

1. Odabasi, A. (2010). A study on laser beam welding technique: Effect of heat input on the microstructural evolution of superalloy inconel 718. doi: 10.1007/s1161-010-0329-y
2. Radhakrishnan, B. (1988). A quantitative microstructural study of intergranular liquation and its relationship to hot cracking. *Metallography*, 21, 453-471.
3. Huang, C. A., Wang, T. H., Lee, C. H., & Han, W. C. (2005). A study of the heat-affected zone (haz) of an inconel 718 sheet welded with electron-beam welding (ebw). *Material Science and Engineering*, 398, 275-281. doi: 10.1016/j.msea.2005.03.029
4. Idowu, O. A., Ojo, O. A., & Chaturvedi, M. C. (2007). Effect of heat input on heat affected zone cracking in laser welded ati allvac 718plus superalloy. *Material Science and Engineering*, 454-455, 389-397. doi: 10.1016/j.msea.2006.11.054
5. Ojo, O. A., Wang, Y. L., & Chaturvedi, M. C. (2008). Heat affected zone liquation cracking in electron beam welded third generation nickel base superalloys. *Material Science and Engineering*, 476, 217-223. doi: 10.1016/j.msea.2007.04.091
6. Thompson, R. G., Radhakrishnan, B., & Mayo, D. E. (1989). Intergranular liquid formation, distribution, and cracking in the haz of alloy 718 welds. *Metallurgy and Applications*, 437-455.
7. Baeslack III, W. A., & Nelson, D. E. (1986). Morphology of weld heat-affected zone liquation in cast alloy 718. *Metallography*, 19, 371-379. doi: 0026-0800/86/\$03.30
8. Kou, S. (2003). Solidification and liquation cracking issues in welding.

Acknowledgments

The author would like to thank the Center for Friction Stir Processing and The National Science Foundation for providing the funding for this research. Thanks to advisors Dr. Bharat Jasthi and REU site director Dr. Michael West for their direction and guidance, Professor of English Dr. Alfred Boysen for his critique in writing and speaking, and a special thanks to all of the faculty and staff at SDSM&T and the students working in the Advanced Material Processing Center (AMP) for their help.