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LIST OF ACRONYMS

ANL: Argonne National Laboratory
ASTM: American Society of Testing and Material
CVD: Chemical Vapor Deposition
EDAX: Energy Dispersive X-ray spectroscopy
FSL: Friction Stir Link
FSP: Friction Stir Processing
FSW: Friction Stir Welding
MPR: Micro Pitting Rig
PCBN: Poly-Crystalline Boron-Nitride
PVD: Plasma Vapor Deposition
SEM: Scanning Electron Microscopy
TEM: Transmission Electron Microscopy

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1. Executive Summary:

Friction at contacting surfaces in relative motion is a major source of parasitic energy loss in machine systems and manufacturing processes. Consequently, friction reduction usually translates to efficiency gain and reduction in energy consumption. Furthermore, friction at surfaces eventually leads to wear and failure of the components thereby compromising reliability and durability. In order to reduce friction and wear in tribological components, material surfaces are often hardened by a variety of methods, including conventional heat treatment, laser surface hardening, and thin-film coatings. While these surface treatments are effective when used in conjunction with lubrication to prevent failure, they are all energy intensive and add significant cost.

A new concept for surface hardening of metallic materials and components is Friction Stir Processing (FSP). Compared to the current surface hardening technologies, FSP is much more energy efficient (>95% reduction in energy usage), has no emissions or waste by products (e.g. quenchant), and has now been demonstrated to be able to achieve equal or better tribological performance than the traditional technologies. If implemented, greenhouse gas emissions should be able to be reduced by an equivalent 95% or more. Furthermore, FSP is a much faster technique with resulting time and cost savings.

FSP involves plunging a rotating tool to a predetermined depth (case layer thickness) and translating the FSP tool along the area to be processed. This action of the tool produces heating and severe plastic deformation of the processed area. For steel, the temperature is high enough to cause phase transformation, ultimately forming hard martensitic phase. Indeed, FSP has been used for surface modification of several metals and alloys so as to homogenize the microstructure and refine the grain size, both of which led to improved fatigue and corrosion resistance.

Based on the potential benefits of FSP, a concept project was proposed to investigate the commercial and technical feasibility of replacing traditional heat and surface treatment process with FSP. In this project, it was expected to have beneficial effects on friction and wear performance of metallic materials. However, little or no knowledge existed on the impact of FSP concerning friction and wear performance – the subject of this project. Specifically for steel, FSP could potentially replace the current conventional surface hardening techniques used for friction and wear performance. Given the wide use of steel for application where tribological properties are important, the potential market is very large.

Friction Stir Link Inc. (FSL) teamed with Argonne National Laboratory (ANL) to develop and optimize FSP for friction and wear performance enhancement. The ultimate goal is to offer FSP and an effective alternative to some of the current energy intensive and high-cost surface hardening processes. In this first phase, FSL developed the FSP technique for 4140 steel; an alloy commonly used in wear applications. ANL conducted the pertinent friction and wear studies to assess the tribological performance of FSP-processed material in comparison to the performance of the same material processed by conventional surface hardening.

In this concept project, both technical and commercial viability of applying FSP to 4140 Steel were investigated. Some of the major findings included:

- Technical Viability

  - FSP can be applied to 4140 Steel, generating a microstructure suitable or consistent with application where tribological properties are important.
  - Hardness of the FSP material met or exceeded that generated from traditional heat treatment processes
  - Wear and friction properties met or exceeded that generated from traditional heat treatment processes. Friction and wear testing was performed in both the uni-directional and reciprocating modes in both lubricated and dry conditions.
  - There are numerous potential applications where parts are in relative motion.
- Commercial Viability and Energy Savings
  - Energy savings depend on the amount (area) that needs to be processed, but energy savings in excess of 95% versus traditional heat treatment processes is achievable.
  - Processing Cost
    - Savings can be quite significant (~75%), if FSP areas are not required over entire part. Many applications do not require entire part to be processes.
    - If large areas need to be processed, then FSP cost can significantly exceed that of the traditional process. FSP cost is primarily driven by the cost of the FSP tool and its relative tool life.
  - Energy savings potential vs. Market – Heat treating in USA alone is estimated to be about $20 -25 billion a year business involving about 20,000 manufacturers. As much as 5-10% of the current heat treated parts are candidate for FSP based surface hardening. This could translate to a potential $100 to 250 million a year market for FSP in surface hardening. Of course, as the technology is optimized and further developed, an increase in market size is expected.
- Technical or Commercial Hurdles
  - Machine control developments are necessary to ensure a stable and robust process
  - A significant percentage of the potential applications will have restricted access. This means, lower force and physically smaller equipment will need to be developed to realize the full market potential.
  - In this concept study, only one alloy was investigated. There are many other alloys that are used in applications where parts are in relative motion and tribological properties are important.
  - To further expand the market or realize its potential, tool life improvements appear necessary.
- Recommendations: The replacement of traditional, energy intensive heat treatment processes with the energy efficient friction stir processing technology has been demonstrated to be technically and commercially viable. However, further development is necessary in order to realize production capability. The following are recommended activities for further development
  - Process optimization to determine if further improvements in tribological properties with FSP can be generated. With this, a greater understanding of the controlling factors or inputs for post FSP tribological properties can be determined. In addition, other enhancements to the FSP process are envisioned which can potentially improve the results beyond what has been demonstrated to-date.
  - Production control systems need to be developed.

- Reductions in processing forces and miniaturization of FSP equipment needs to be performed to allow FSP to be viable in the many applications with restricted access.
- FSP processes in other steel alloys used in these applications needs to be developed.

2. Project Objective:

The main objective of the current proposed 1.35 year effort was to demonstrate friction stir processing (FSP) as a potential new (next generation) manufacturing concept for surface hardening so as to improve friction and wear performance of tribological components. This was done by applying FSP to steel alloy 4140 (typical tribological component material), conducting laboratory bench-top friction and wear performance evaluations, and comparing results with performances of surfaces subjected to the conventional hardening treatment. Both the FSP processing and friction and wear studies included detailed mechanistic studies to provide the basis for process optimization and extension to other materials during the follow-on work.

The proposed concept can potentially be used to supplant traditional, energy intensive, surface hardening processes, such as furnace heat treating, induction hardening, laser surface hardening etc. FSP is a much more energy efficient process compared to these other processes. Furthermore, hardening may only need to be applied locally, allowing FSP to show further energy reductions. In addition to being a replacement for surface hardening, it may be possible to improve friction performance above and beyond traditional hardening processes. If this benefit is realized, reductions in energy usage should be achievable in manufactured product with components in relative motion (e.g machinery, vehicles, etc.)

3. Background:

Friction stir processing has previously been demonstrated to locally modify material properties. Depending on the material and alloy, FSP has been shown to have positive effects on ductility, fatigue properties, fracture toughness, among others. However, there has been limited to no work investigating the ability of FSP to harden or improve friction and wear properties of materials used in applications where parts are in relative motion. One common material used in these applications is 4140 steel.

If FSP could be used to locally improve wear properties, then use of typical energy intensive manufacturing processes could be avoided. If these properties could be improved beyond the capability of the traditional processes, further energy reductions could be realized in the end-product.

Based on the potential of FSP to improve friction and wear properties, this project was proposed to investigate the technical and commercial feasibility of the concept. The specific goals included

1) Assess technical viability of FSP of alloy 4140 steel.
2) Assess technical viability of FSP to improve wear and friction properties of 4140 steel vs. traditional heat treatment (HT) processes..
3) Assess the manufacturing viability of FSP of alloy 4140 steel.
4) Assess the commercial viability of FSP of alloy 4140 steel. This specifically involves
    a. Identify potential applications
    b. Assessing manufacturing cost in potential applications vs. traditional HT process.
    c. Assess potential energy reductions in identified application, if FSP is implemented

5) Identify opportunities for optimization and further research and development needed to complete commercialization process

4. Accomplishments:

- During the first quarter the following accomplishments were made
  - Initial FSP trials were successfully completed on 4140, which were used for initial material property modification analysis and to define a more detailed secondary set of FSP trials
  - Initial trials of FSP on AISI 4140 steel showed significant hardening of the process surface layer through the formation of martensitic phase
  - Based on original trials, a test plan was established for secondary FSP trials.
  - Initial plans towards application identification and commercial viability assessment were completed.
- During the 2nd quarter, the following accomplishments were made
  - Secondary FSP trials were successfully completed on 4140. The essential FSP variables which were investigated included
    - Multiple FSP Tools
    - Trials over a range of travel speeds, rotation speeds, and processing force
    - Single and multiple pass trials
  - Cross-sectioning, tensile testing, and hardness testing was initiated on secondary FSP trials samples.
  - Secondary trials of FSP on AISI 4140 steel confirmed significant hardening of the process surface layer through the formation of martensitic phase
  - Progress towards application identification and commercial viability assessment were completed.
- During the 3rd quarter.
  - The samples generated in the 2nd quarter were subsequently used for material property modification analysis in an effort to determine if there was any correlation between the essential FSP variables and subsequent material properties.
  - Cross-sectioning and hardness testing was completed on secondary FSP trials samples of 4140 steel.
  - Hardness data and cross-sections were analyzed to determine potential correlation between results and
    - FSP Tool Design
    - Travel speeds, rotation speeds, and FSP pitch or relative heat input
    - Single and multiple pass
  - Secondary trials of FSP on AISI 4140 steel confirmed significant hardening of the process surface layer through the formation of martensitic phase
- During the 4th quarter, the following progress was made
  - Commercial viability analysis was completed to 80% level.
  - Test bed system details drawings were completed. Test bed system will be used to assess manufacturing viability on typical production equipment.

  - Test bed system fabrication was started with about 60% completion as of June 30.
  - Completed tribological performance evaluation of FSP processed 4140 steel in unidirectional sliding condition under dry contact.
  - Started tribological performance evaluation in reciprocating contact under dry contact conditions.
- During the last period, the following progress was made
  - Commercial viability analysis was completed.
  - Test bed system fabrication was started and completed.
  - Completed tribological performance evaluation in reciprocating contact under dry contact conditions.

5. Results & Discussion:

5.1 Task 1.0: Potential Benefits Assessment

For potential applications that were identified during the 3rd quarter, commercial feasibility and energy savings analysis was completed for one of the applications (bearing race shown in Figure 1), given it is representative of many applications.

Figure 1: Bearing Race Application

The following key assumptions were made as part of this analysis

1) Part:
   a. 18" diameter, with 3" wall
   b. 6" height.
   c. Alloy 4140.
2) Heat Treatment to Rc 55 to 60.
   a. Cost of $133/hr.
   b. Total time of 6.5 Hrs.
   c. Heat treatment to 55 to 60 Rc requires.
   d. Total energy use of 700 kilowatt-hours was assumed, based on natural gas consumption rate provided by heat treatment supplier.
3) Carburizing

a. Cost of $133 / hr.
b. Carburizing to .020" depth requires 9 hours, including tempering time. Assumes .004"/hr diffusion rate.
c. Total energy use of 900 kilowatt-hours was assumed, based on natural gas consumption rate provided by heat treatment supplier.
d. Other Notes:
    i. Mostly applicable to low carbon steels.
    ii. For higher carbon content steels standard heat treatment is normally used.
    iii. Applicable to case hardening depths up to about 0.25"
    iv. Need for post processing (e.g. grinding to eliminate distortion or provide improved surface finish) will help dictate desired depth and associated cost.

4) Nitriding
    a. Cost of $80 / hr.
    b. Gas Nitriding to a depth of .02" (.5 mm) requires 30 hrs. This equates to a diffusion rate of approximately .0006" / hr
    c. Total energy use of 2000 kilowatt-hours was assumed, based on natural gas consumption rate provided by heat treatment supplier.
    d. Other Notes:
        i. Applicable to case hardening depths up to about 0.02 to .025 inches. This is significantly shallower than carburizing.
        ii. Distortion significantly less than other processes.
        iii. Need for post processing (e.g. grinding to eliminate distortion or provide improved surface finish) may eliminate nitriding as an acceptable case hardening process
5) Friction Stir Processing
    a. FSP Tool cost of $3000 / tool
    b. Machine cost of $125/hr (exclusive of FSP tool)
    c. Tool life of 300 feet
    d. Power requirement of 3 Hp (2.2kw), based on actual data.
    e. Travel speed of 4 inches per minute
    f. Assumes that the ENTIRE surface is processed.

Calculations of energy and cost savings were done for the bearing race application. Given the notes above, it is emphasized that the steel alloy and the application characteristics (i.e. desired depth of hardening, sensitivity to distortion) will affect the decision on what process to use. For simplicity and comparison purposes, it is assumed that each of these processes would be applicable to the current alloy, with knowledge that this is not likely true. The main purpose of this analysis is to determine relative cost of each process versus FSP and to determine potential energy requirement reductions. The costs were calculated for a typical batch size of the example component. It is also noted that heat treatment costs tend to be highly correlated with batch size, primarily below the level of a full furnace load.

The equations and formulas used to calculate the energy requirements and costs for both alternatives are indicated in the Appendix.

Given the inputs above, the following summarizes the results

- Energy Savings
  - FSP requires 93% less energy than heat treating
  - FSP requires 95% less energy than carburizing
  - FSP requires 98% less energy than nitriding
- Direct cost of 100% FSP (full surface is friction stir processed) vs. each process
  - FSP cost is 5 times more costly than heat treating
  - FSP cost is 4 times more costly than carburizing
  - FSP cost is 2 times more costly than nitriding
  - FSP tool cost is 70% of total cost with the parameters above.
- If FSP could be limited to a single pass, which is possible in many applications, FSP is less costly in all cases and could save as much as 75% vs. nitriding.
- Other notes
  - Actual (future) FSP cost will likely decrease dramatically for the following reasons
    - FSP cost is directly proportional to the amount of FSP. Applications where single pass or fewer passes would be required directly reduce cost.
    - FSP cost is inversely proportional to tool life. Since early FSW/FSP development, tool life has increased by at least an order of magnitude. There is no reason to suspect the trend will not continue.
    - FSP cost is inversely proportional to travel speed. FSP of steel is early in its development stage. FSW of steel with greater penetration have demonstrated travel speed increases of factors of 3 or 4. It is anticipated that similar improvements could be realized with this application.
    - FSP tools for steel are still only produced in low volume. As the number of applications for FSP/FSW of steel increase, the volume of tools will increase, driving tool cost lower.
    - The depth of processing maybe reduced in many applications without reduction in performance. This will allow for tool life improvements and travel speed increases. Current trials have produced depth of processing significantly in excess of what is typically performed with other case hardening processes.
  - The costs above did not consider other costs such as costs to replace a worn out component. The Timken bearing race for the windmill is a prime example. This application is currently demonstrating bearing life that is too low. The cost to replace the bearings is orders of magnitude higher than the processing cost. Thus, there are applications where performance (or cost of non-performance with traditional technology) is the deciding factor, not direct processing cost of the new technology.
  - It is anticipated that the relative cost comparison will generally be applicable to most applications, regardless of the size of the part. Cost of FSP and any heat treatment or case hardening process is generally proportional to the size of the part. This is because a smaller part would require less FSP. Furthermore additional smaller parts can be placed in the same furnace, lowering the cost / part.

- The items noted above which would lead to FSP cost reductions are areas of opportunity and are recommended to be areas of further research.

5.2 Task 2.0: FSP Development for 4140 Alloy Steel

FSW/FSP of steels require use of special tool materials and process control. To develop some initial information to guide future periods of the project, early in the project a visit was made to a FSP tool supplier (MegaStir, Inc.) to perform an initial feasibility trial and to develop knowledge to enable a technology transfer process. An initial sample was made in ¼” thick plate on a laboratory level machine and setup. A single FSP tool was used over a small range in FSP parameters. The material was successfully processed. However, the necessary control of the process indicated that the secondary FSP trials should also be performed at the tool supplier for risk reduction purposes and project timing.

Plans were made for the secondary FSP trials. There were multiple objectives of the secondary trials. These included

1) Varying tool design and critical FSP parameters to establish any parameter effects and opportunities for optimization for the purpose of development of an initial understanding of the process envelope and effect of the essential variables. The essential variables include travel speed, rotation speed, processing force, FSP tool design, and FSP tool material.
2) Determine significance of wear of FSP tool. FSP tool are costly, so tool life is an important parameter in determining process cost.
3) Perform an initial assessment on the viability of multi-pass FSP, so as to determine viability to process larger areas.
4) Further assess needs for transferring technology to a manufacturing environment. Note all these trials will be performed on a laboratory level system. The objective of this subtask is to define critical needs for a production capable FSP machine for technology transfer reasons.

For these trials, FSP tools and steel were ordered, and final plans made for these secondary trials at the end of the first quarter. To perform these secondary FSP trials a return visit was made to a FSP tool supplier (MegaStir, Inc) to perform the secondary FSP.

FSP trials were conducted with greater than 30 different settings of the essential variables. In addition, some trials were repeated to aid in developing an understanding of the robustness of the process. A photograph during processing is shown in Figure 2. The samples were all to be subjected to cross-sectioning, tensile testing, and micro-hardness testing.

The following observations and findings resulted from the post-FSP analysis

1) Cross-sectioning indicated a range of post processed region characteristics from fully-consolidated to existence of internal voids. Figure 3 and Figure 4 show the range.
2) Visual observations indicate that FSP tool design has a significant effect on the resulting processed region.

3) The area of acceptable FSP parameters (rotation speed, travel speed) appears to be dependent on the tool design. This is similar to the friction stir welding process.
4) There was not excessive tool wear.
5) Multi-pass FSP did not appear to cause an increase in tool wear and appears to be feasible. Multi-pass FSP requires reprocessing of area that will have been processed (and hardened) by previous passes. A photograph of a section of plate that has been processed in a multi-pass fashion is shown in Figure 5.
6) Hardness testing results indicated little to no correlation between resulting hardness vs. travel speed or rotation speed as shown in Figure 6, which displays the average Vickers hardness and maximum Vickers hardness over width of the test region (to approximately 15 mm on each side of the centerline of the FSP). The measurements were made at a depth of 1 mm from the top and with a spacing of approximately 0.5 mm. However, it can be seen that there is a significant difference in the resulting hardness vs. the FSP tool designed.
7) As noted, hardness testing results indicated that there exists a correlation between the tool design and resulting hardness. To further understand the effect of each tool, the data for the two tools with FSP trials at the same travel speeds and rotation speeds were averaged over all samples and plotted vs. the distance from the center line. This is shown in Figure 7. It can be seen that the second tool yields higher Vickers hardness and a wider zone of increased hardness. It is noted that the overall width (diameter) of the FSP tools are the same, but the pin diameter of the second tool is larger.
8) Multi-pass samples were also cross-sectioned. It can be seen that the FSP region is fully consolidated across the width of the FSP region, indicating that it is feasible to process a larger region than possible with just a single pass. This is shown in Figure 8.
9) Vicker's Hardness profiles were also measured on the multi-pass samples, across the width direction (shorter dimension). The resulting data is shown in Figure 9. It can be seen that the highest Vicker's hardness values are on the exterior and that the hardness values in the center region are lower than for the single pass FSP. This indicates the annealing affect of the subsequent passes on the previous FSP passes.

Figure 2: Friction Stir Processing of 4140 Steel

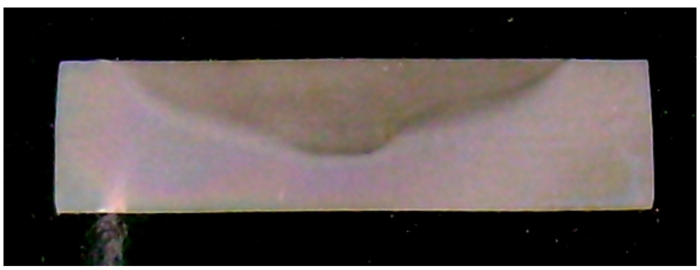


Figure 3: Macro picture of Fully Consolidated FSP Region

Figure 4: Macro picture of FSP Region with lack of consolidation

Figure 5: Multi-Pass FSP Raster Pattern

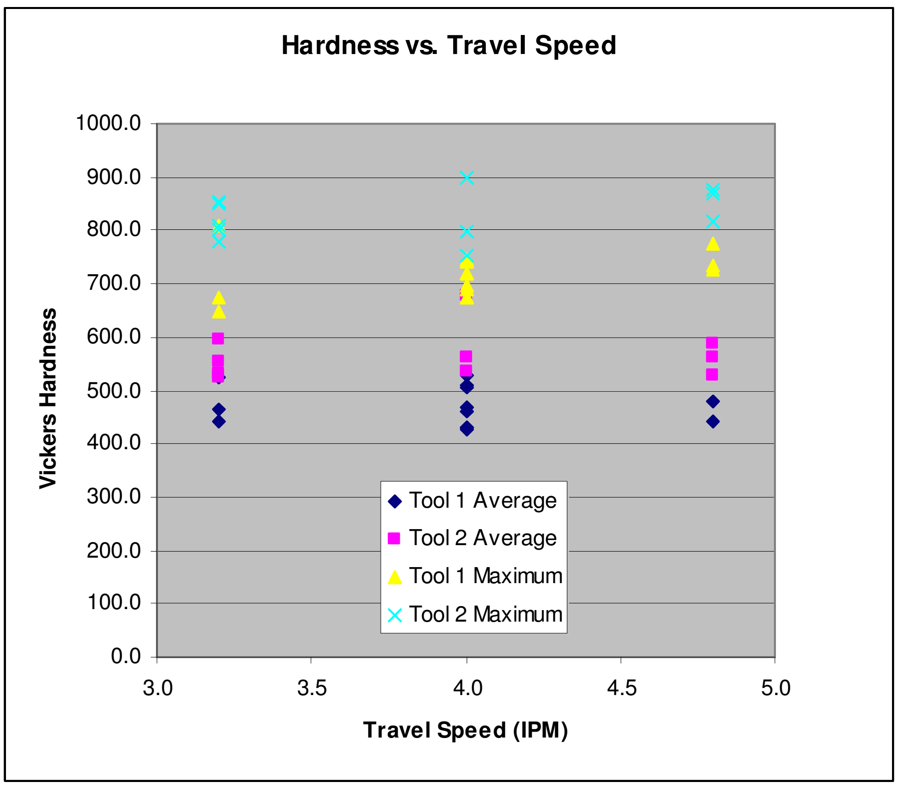


Figure 6: Average and Maximum Hardness of FSP Region vs. Travel and Rotation Speed

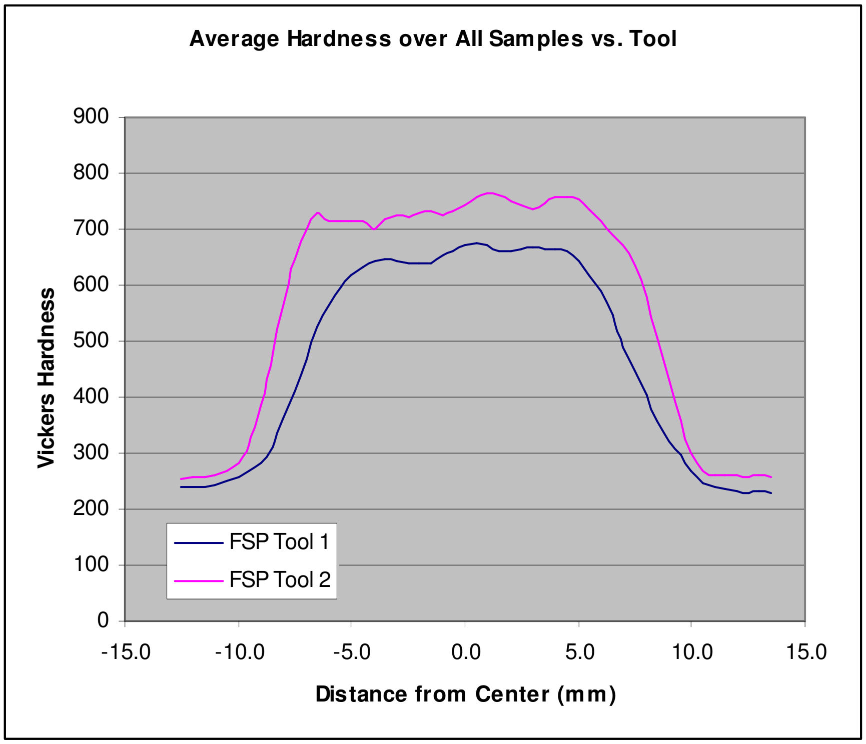


Figure 7: Average Hardness Data for the Two FSP Tool Designs

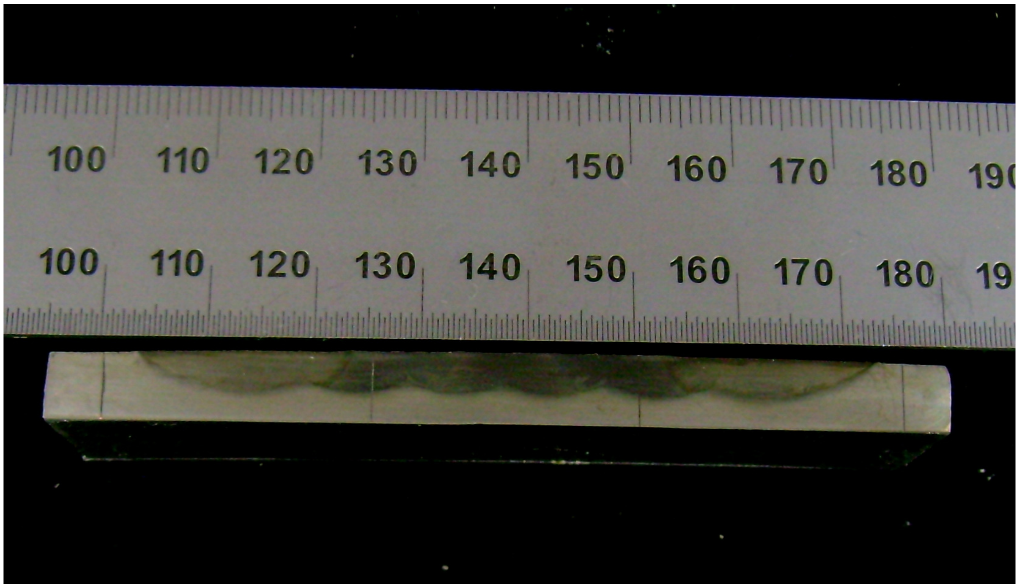


Figure 8: Multi-Pass FSP Cross-Section

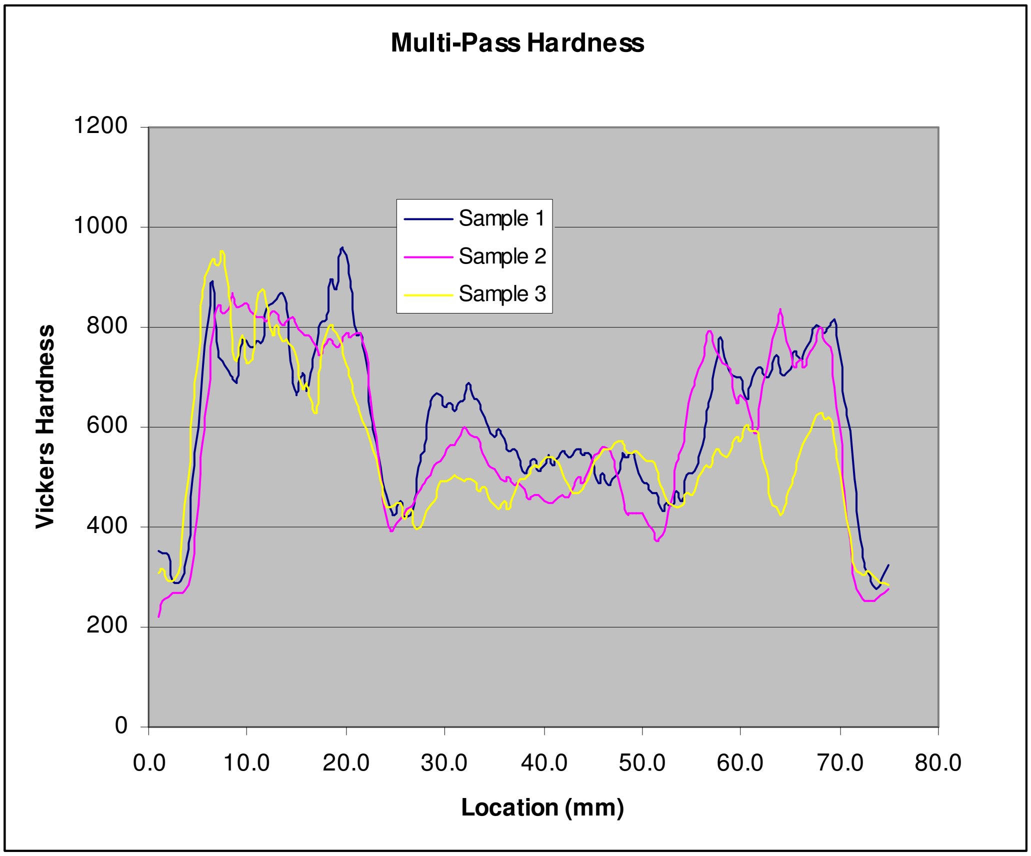


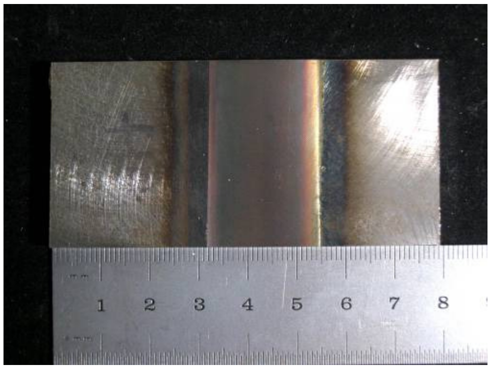
Figure 9: Multi-Pass Vicker's Hardness

Based on the lessons learned from the initial and secondary trials, plans and specifications for a test bed system were completed. The purpose of the test bed system was to provide an initial assessment of the viability of the FSP process on equipment more typically used for friction stir welding production. After determining the specifications, design work was initiated and completed. By the end of the project the test bed system was assembled and initial test and debug started. The initial test and debug indicated positive results, however project funding did not allow for extensive testing to be completed.

5.3 Task 3.0 Microstructural and Tribological Performance Evaluation of FSP

5.3.1 Microstructural Evaluation

A segment of the initial FSP trial on the 4140 steel plate shown in Figure 10 was provided to ANL for microstructral, mechanical and tribological properties evaluation. Conventional cross-sectional metallography was conducted on the FSP plate. Mounted samples were polished and etched with 2% Nital. A macro-picture of the etched sample is shown in Figure 11. The processed area with the martenstic phase transformation can be clearly seen. Optical microscopy showed that the original microstructure consists of pearlite (left side of Figure 12) as expected for annealed 4140 steel. The microstructure of the processed area consists of martensite (right side of Figure 12)



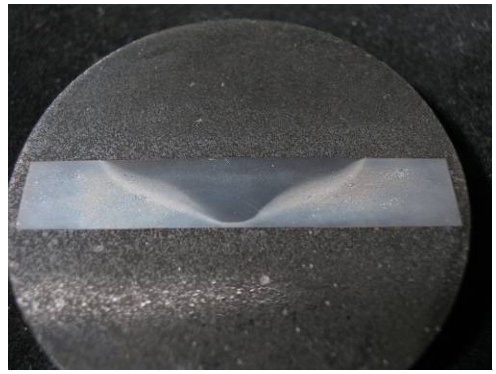


Figure 10: Macro picture of FSP plate **Figure 11: Macro picture of etched sample**

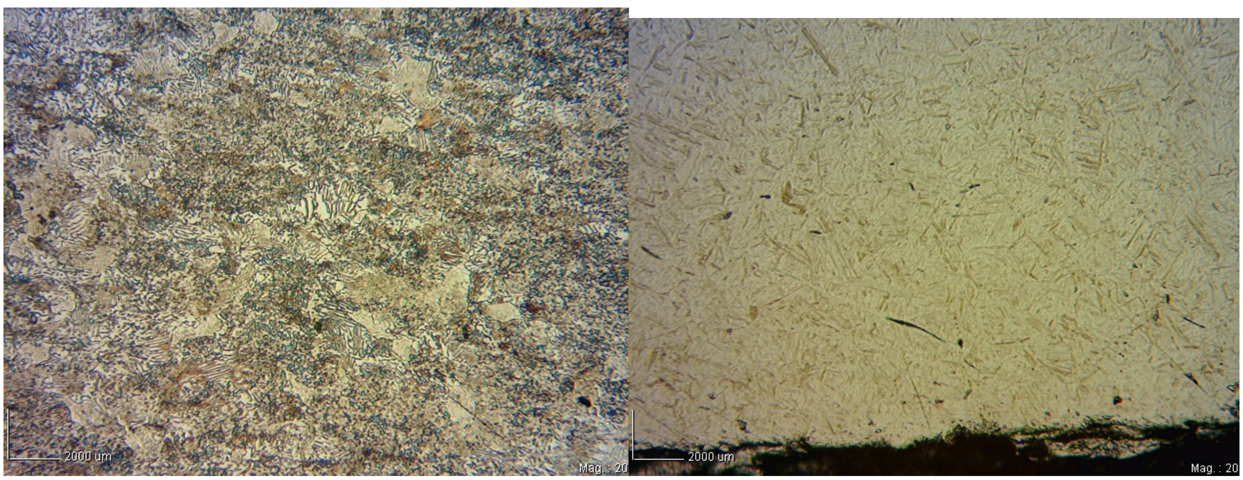


Figure 12: Pre-FSP microstructure (pearlite) and Post FSP microstructure (martensite)

Micro-hardness measurement with Vickers indenter at a load of 1kg showed that the baseline material (pearlite) has a hardness of about 200VHN, while the hardness of the FSP processed area is about 594 VHN (58.6R_c). This represents a significant hardening, comparable to the case carburization for the same material. A piece of the baseline 4140 steel was also subjected to conventional heat treatment. The sample was austenitized at 850°C for 1 hour, and then water quenched. The microstructure of heat hardened material consists of martensite and the measured microhardness was about 450VHN (45.7R_c). These results showed that FSP produced the same microstructure in terms of phase and significantly higher surface micro-hardness compared to conventional heat hardening, perhaps reflective of grain refinement by FSP.

Test samples were fabricated for friction and wear performance evaluation of the baseline, heat hardened and FSP materials.

5.3.2 Reciprocating Contact Friction and Wear Testing Equipment and Parameters

Friction and wear tests were conducted with a ball-on-flat contact configuration in reciprocating sliding. Figure 13 shows the picture of the test rig and typical specimen contact.

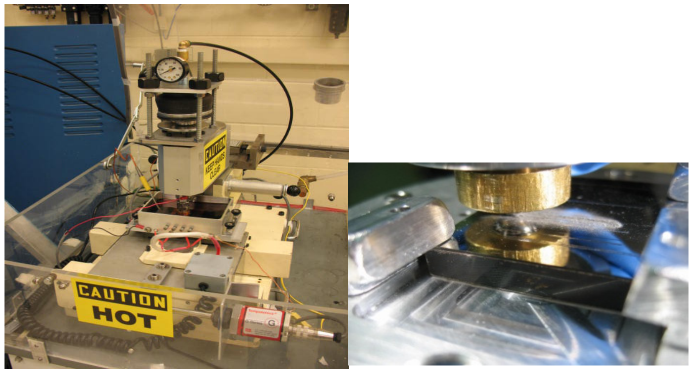


Figure 13: Reciprocating ball on flat friction and wear test rig

Ball specimens are made of polished 12.7 mm (0.5") diameter hardened 52100 bearing steel with a surface roughness of 0.035 μm Ra. Rectangular flat specimens with 50 x 30 x 0.25 mm nominal dimensions were made from 4140 steel in four different conditions: the annealed baseline, heat treated (HT) water quenched, HT salt quenched and FSP. The surfaces of all the flat specimens were ground to the same surface finish of 0.14 μm Ra.

Tests were conducted at three different loads of 25, 50 and 75N which impose Hertzian contact pressure of 0.80, 1.0, and 1.15 GPa, respectively, for a ball-on-flat contact. Reciprocating frequency of 1 Hz was used, with a stoke length of 10 mm, translating to an average sliding velocity of 1 cm/s. Test duration was 1 hour and under ambient room temperature. All the tests were lubricated with basestock synthetic poly-alpha-olefin (PAO4) oil containing no additives and with a viscosity of 18 cSt at 40˚C.
Friction coefficient was continuously measured during each test. At the conclusion of the test wear was measured in the ball and flat test specimens by optical profilometry technique. Worn surfaces were also characterized by both optical and SEM microscopy to assess wear mechanisms.

5.3.3 Results: Friction and Wear Testing in Reciprocating contact.

Figure 14 shows the variation of friction coefficient for the duration of the test with the various flat specimen. Friction behavior is essentially the same for all the materials at the three different loads tested. The magnitude of friction coefficient is between 0.1 and 0.12; which is typical for boundary lubrication regime. The friction of the baseline material is also noisier compared to the hardened surface, perhaps indicative of more local plastic flow at the asperity contact.

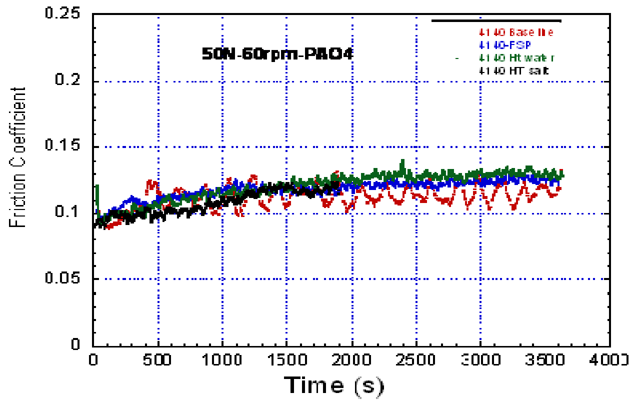


Figure 14: Variation of friction coefficient with time at normal contact load of 50 N

Wear in each flat specimen at the conclusion of the test is shown in Figure 15. Although, the friction behaviors were similar for all the flats, wear behaviors were very different. Significant wear reduction was observed in all the hardened surfaces compared to the baseline material. FSP reduced the wear most of all the three hardened techniques. In the FSP specimens tests were run with the reciprocating sliding direction perpendicular (FSP⊥) and parallel (FSP//) to the tool translation direction. From Figure 15, the least amount of wear was observed when sliding in the parallel direction. It should also be noted that the amount of wear increases with increasing load, but much less so in the FSP material, especially when sliding in the parallel direction.

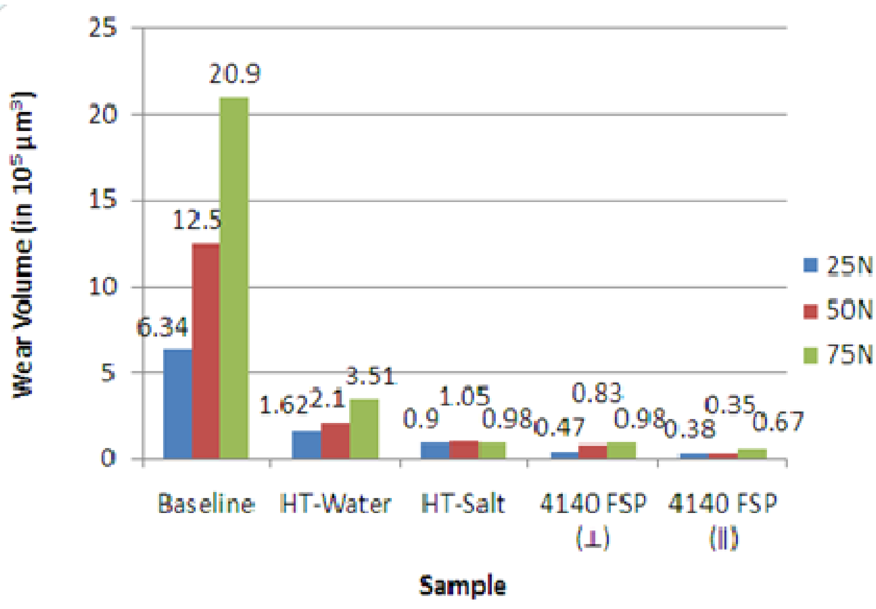


Figure 15: Wear in the various 4140 steel flats at the three test loads

Examination of the worn surface showed that wear in the baseline material involves significant amount of plastic deformation as indicated by material pile-up at the edge of track in Figure 16. SEM analysis indicates the wear mechanisms in the baseline material consist of abrasion, as indicated by scratches in the direction of sliding and fatigue indicated by cracks formation and material loss. In heat hardened flats, less plastic deformation occurred. There is no evidence of material pile-up at the edge and material removal occurred primarily by abrasion. The increase in hardness is expected to reduce the amount of plastic deformation, which also reduces the extent of fatigue damage. Nonetheless, considerable amount of material removal did occur.

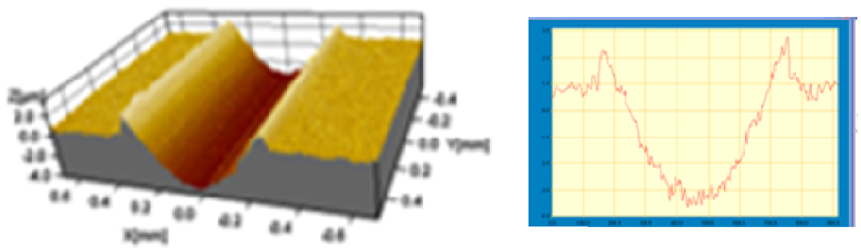


Figure 16: Optical 3-D and 2-D profilometry of wear track in baseline 4140 steel

In the FSP flat, minimal surface damage and material removal occurred. There were only a few scratches and minimal plastic deformation (Figure 17). Indeed SEM analysis show occurrence of only superficial damage and material removal. An original grinding mark running across the wear track can still be clearly seen in figure 17; illustrating minimal material removal.

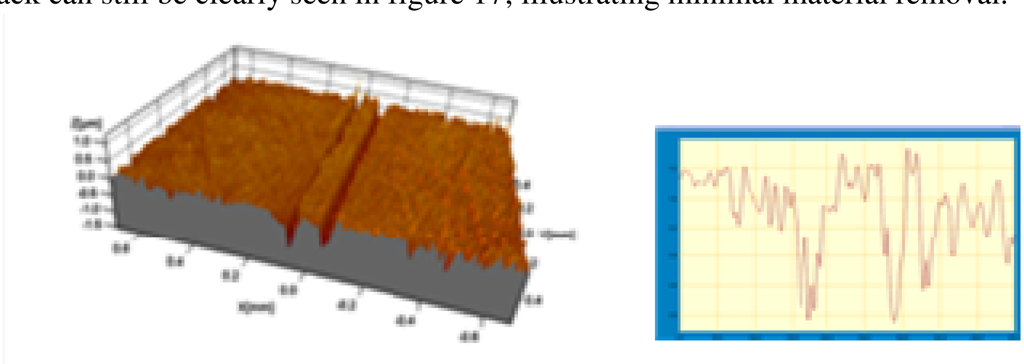


Figure 17: Optical 3-D and 2-D profilometry of wear track in FSP 4140 steel flat

Results of the study of the initial samples showed that FSP has an excellent potential to enhance the wear resistance of 4140 steel. Compared to the baseline material, FSP reduced wear by more than a factor 20 and a factor of 2 compared to traditional heat hardening treatment. The wear mechanism was also changed from abrasion and fatigue to a superficial surface damage by FSP. This improvement in wear behavior can be attributed to microstructural changes in the near surface material as a result of FSP; consisting of phase transformation and grain refinement.

More friction and wear performance of FSP 4140 steel flats using different variables of rotation and travel speeds and tool designs in lubricated sliding contact with 52100 steel balls were then evaluated. The variations of friction coefficient with test time for various FSP plates are similar to one and other. The average friction coefficient at different normal loads of 25, 75, and 150 N is shown in Figure 18. The average coefficients for the different variants of FSP treatment are similar to one another and the conventional heat treatment. There are four variants of FSP with slightly higher friction coefficients, namely FSP 1-14, 1-16, 2-7 and 2-9. It should be noted that even the higher friction in these four groups is still within the range of typical friction coefficients under boundary lubrication regime. The wear in various flat specimens at the conclusion of the test is shown in Figure 19. The amount of wear in the FSP treated flat is substantially reduced compared to the untreated baseline material. When compared to the heat treated samples, amount of wear in the FSP flats is either similar or lower, except for FSP 2-7 and 2-9 in which wear is slightly higher. This indicates that there maybe an effect of FSP parameters on resulting friction and wear properties. This implies that there may be opportunities to optimize resulting friction properties via modification of FSP parameters and tool designs.

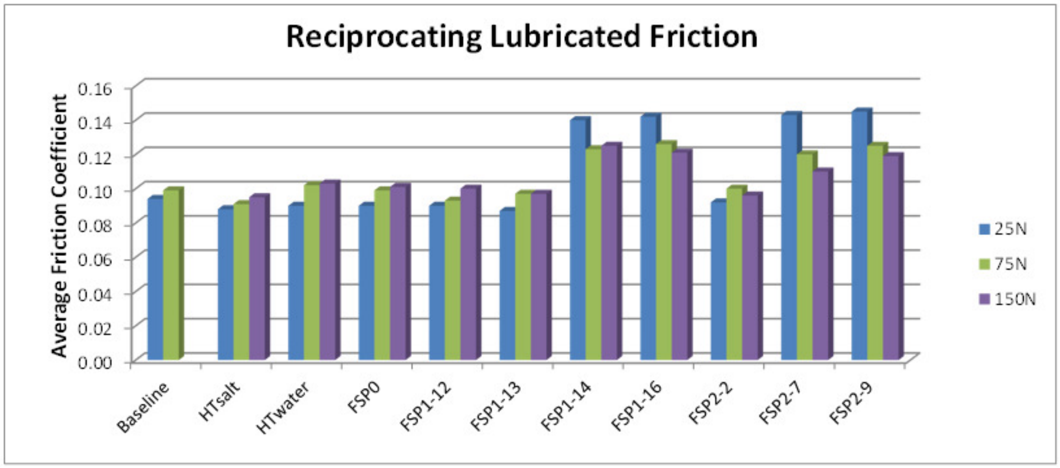


Figure 18: Avg. friction coefficient for lubricated reciprocating 52100 balls & FSP 4140 steel

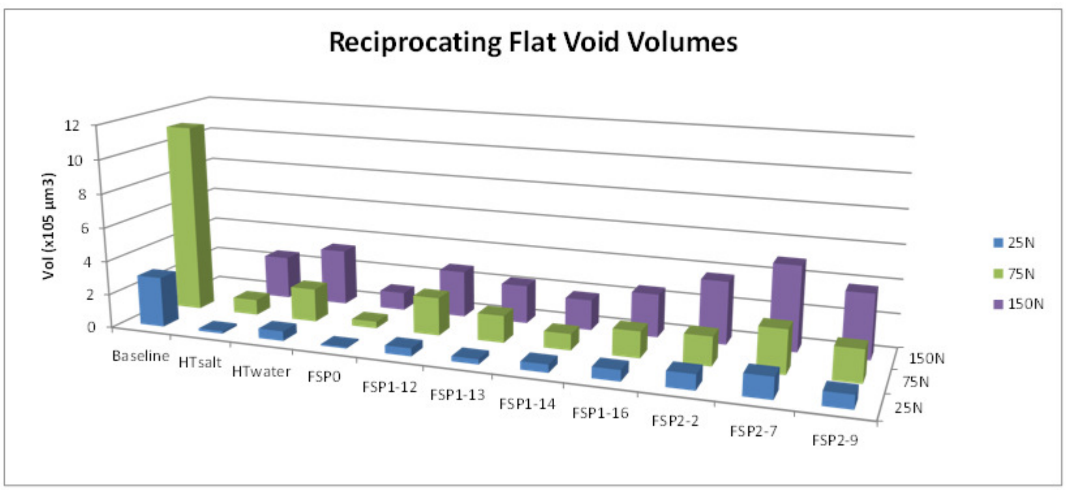


Figure 19: Wear volume after lubricated reciprocating sliding wear test

Friction and wear tests were also conducted with 4140 FSP flats subjected to multi-pass treatment using the standard ball-on-flat contact test configuration. Details of the test procedure are the same as those reported above. The tests were conducted at three different loads of 25, 75 and 150 N and lubricated with base-stock synthetic lubricant (PAO-4). Figure 20 shows the comparison of average friction coefficient for the single pass and three different variants of multi-pass FSP treatments; same direction multi-pass, reverse direction multi-pass, and multi-direction multi-pass.

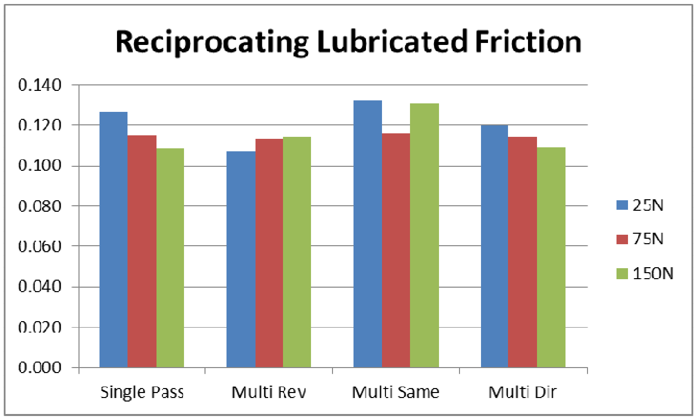


Figure 20: Average Friction in reciprocating lubricated contact

For the three test loads, the average friction in single pass and all the multi-passes are comparable. Comparison of average ball and flat wear is also shown in Figure 21. Multi-pass FSP treatment reduced the average ball wear for the three test loads (Figure 21a). In the flat specimen, some variant of multi pass increased wear, others have no effect (Figure 21b). Although more work is clearly needed to elucidate the impact of number of FSP passes on the friction and wear performance of material, results from the current preliminary test indicate no detrimental effect.

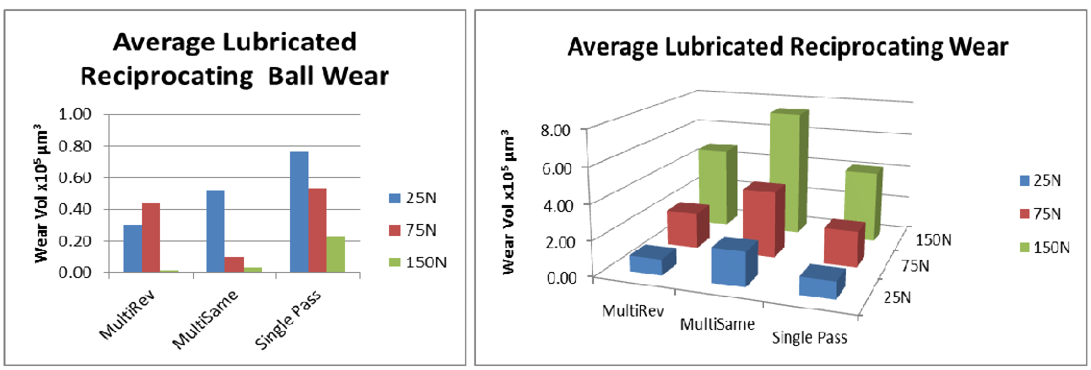


Figure 21: Average wear in reciprocating lubricated contact: a) Ball wear and b) Flat wear

5.3.4 Friction and Wear Testing Equipment and Parameters for Unidirectional Sliding Contact (Lubricated and Dry)

Friction and tests were also conducted in unidirectional sliding contact test using the pin-on-disc tribometer shown in Figure 22. A ball-on-flat contact geometry was used.

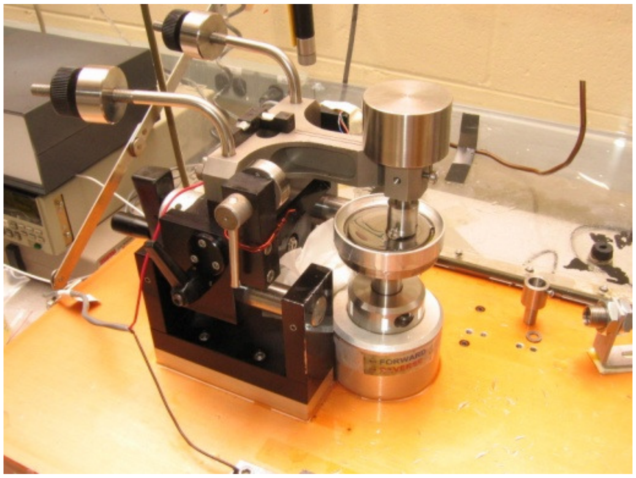


Figure 22: Pin-on-Disc tribometer used for unidirectional sliding friction and wear testing

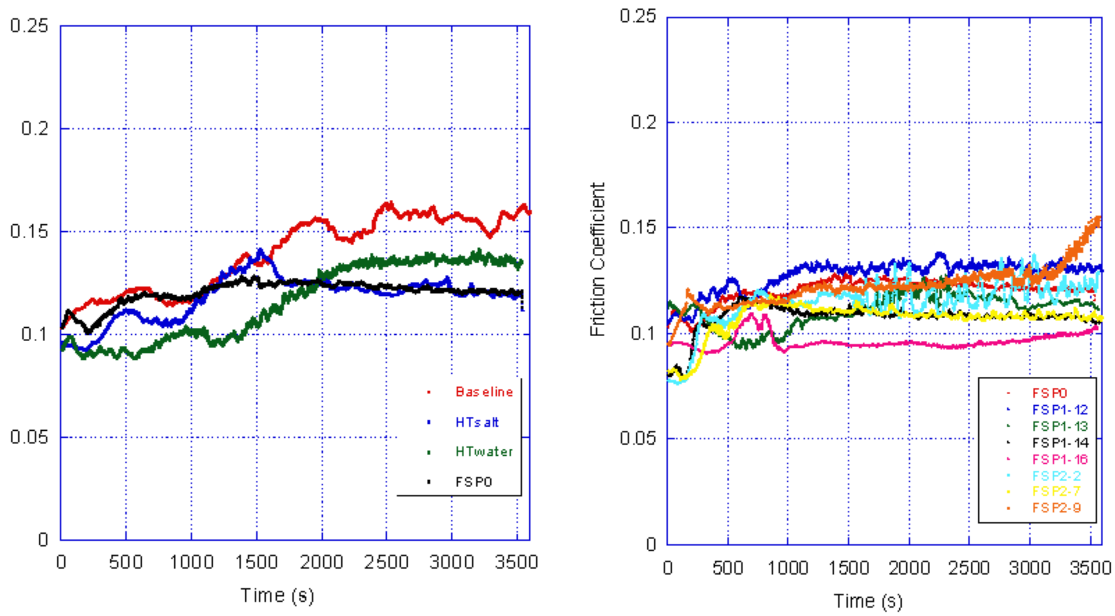
Ball specimens are made of polished 12.7 mm (0.5") diameter hardened 52100 bearing steel with a surface roughness of 0.035 μm Ra. Rectangular flat specimens with 50 x 30 x 0.25 mm nominal dimensions were made from 4140 steel in four different conditions: the annealed baseline, heat treated (HT) water quenched, HT salt quenched and different variants of FSP. The surfaces of all the flat specimens were ground to the same surface finish of 0.14 μm Ra.

Tests were conducted 20 N which impose Hertzian contact pressure of 0.80 GPa, for a ball-on-flat contact. Tests were conducted at a constant sliding velocity of 1 cm/s for duration of 1 hour and under ambient room temperature. For tests with lubrication, all the tests were lubricated with basestock synthetic poly-alpha-olefin (PAO4) oil containing no additives and with a viscosity of 18 cSt at 40˚C.

Friction coefficient was continuously measured during each test. At the conclusion of the test wear was measured in the test specimens by optical profilometry technique. Worn surfaces were also characterized by both optical and SEM microscopy to assess wear mechanisms.

5.3.5 Results: Friction and Wear Testing in Unidirectional Lubricated Contact

Figure 23 shows the variation of friction coefficient for the duration of the test with the various flat specimens. For the baseline material, the friction coefficient increased gradually from an initial value of 0.10 to a near steady value of about 0.16 (Figure 23a). Samples that were heat treated and water quench showed similar trend in behavior, but smaller magnitude with test starting with 0.08 and increasing gradually to about 0.13. For the initial FSP trial treatment, the friction coefficient ranged from 0.11 to about 0.12 for the duration of test and much less noisier compared to baseline and heat hardened specimens. The friction variation for all the different variants of the FSP treatments evaluated thus far is shown in Figure 23b. The friction trends are similar with fiction coefficient values ranging from 0.09 for FSP1-16 to 0. 13 for FSP 1-12, value which are typical for boundary lubrication regime. These results will suggest optimization of FSP processing for a desired frictional behavior or values maybe possible. A summary of the average friction coefficients for the various flat specimens tested is also shown in Figure 24. The results show that friction performance of FSP treated surfaces is comparable or better than those of surfaces heat treated by the conventional method.



(a) Comparison of FSP with other treatment (b) Comparison between FSP variants

Figure 23: Variation of friction with time during unidirectional sliding

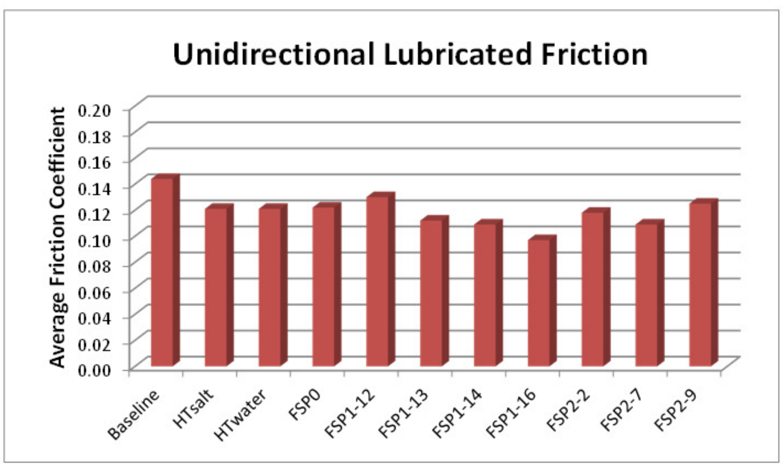


Figure 24: Average unidirectional sliding friction for different flat surfaces

Wear in each flat specimen at the conclusion of the test is shown in Figure 25. All the surface hardening processes substantially reduced the wear in the 4140 steel flat. The wear in the various variants of FSP treatment is comparable to the wear in the traditionally surface hardening treatment.

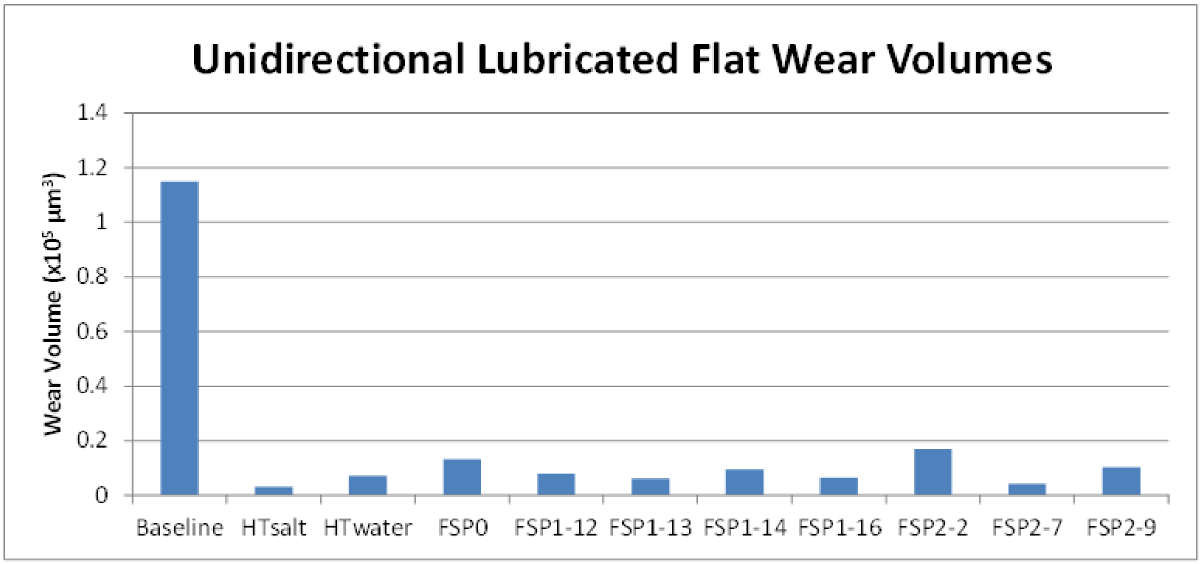


Figure 25: Wear in 4140 steel flats with various treatments

5.3.6 Results: Friction and Wear Testing in Unidirectional Dry Reciprocating Contact

Friction and wear performance of FSP 4140 steel flats with different heat treatment including FSP in dry sliding contact with 52100 steel balls were evaluated. Tests were conducted with baseline, heat treated (water and salt quenched), and FSP1-14, and 2-2 FSP trials. Tests were conducted with normal load of about 5N, reciprocating speed of 1Hz and a stroke length of 1 cm (linear speed 2 cm/s) for duration of 30 minutes.

The variations of friction coefficient with test time for dry sliding tests for various flats are shown in Figure 26. The friction of hardened flat by either heat treatment of FSP is higher than the unhardened baseline material. The two variants of FSP tested thus far showed similar friction behavior with friction coefficient higher than heat treated surfaces. The wear in various flat specimens at the conclusion of the test is shown in Figure 27. The amount of wear in the FSP treated flat is comparable to the other heat treated flats. Wear in the two variants of the FSP tested were about the same; hence only one is shown in Figure 27.

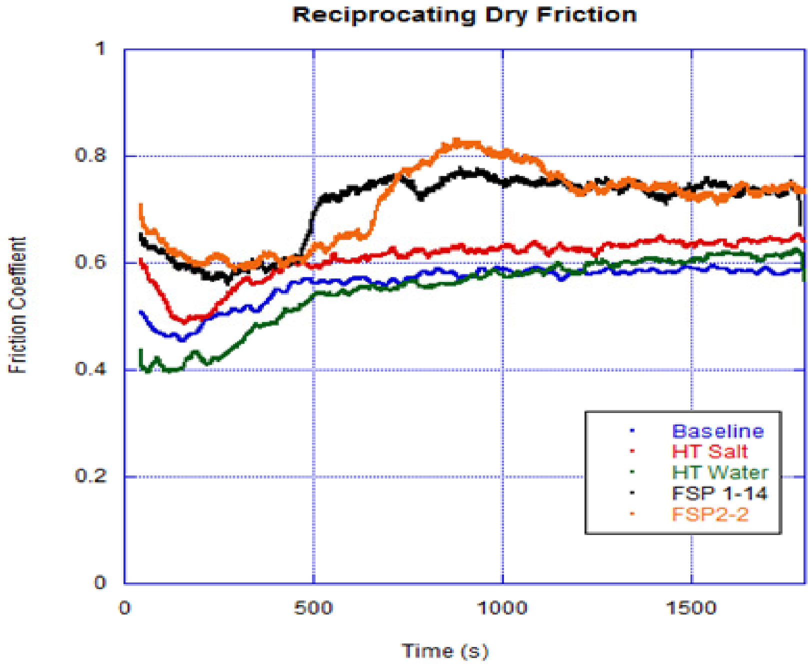


Figure 26: Variation of friction with time during dry reciprocating sliding

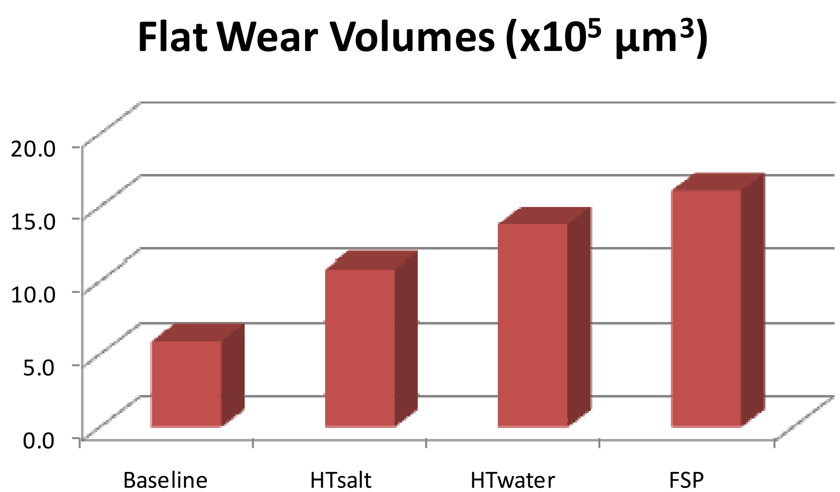


Figure 27: Wear volume in the various 4140 steel flats after dry reciprocating sliding

5.3.7 Results: Friction and Wear Testing in Unidirectional Dry Sliding Contact

Friction and tests were also conducted in unidirectional sliding contact under dry condition using the pin-on-disc tribometer. A ball-on-flat contact geometry was used. Ball specimens are made of polished 12.7 mm (0.5”) diameter hardened 52100 bearing steel with a surface roughness of 0.035 µm Ra. Rectangular flat specimens with 50 x 30 x 0.25 mm nominal dimensions were made from 4140 steel in four different conditions: the annealed baseline, heat treated (HT) water quenched, HT salt quenched and different variants of FSP. The surfaces of all the flat specimens were ground to the same surface finish of 0.14 µm Ra.

Tests were conducted 5 N, constant sliding velocity of 1 cm/s for duration of 1 hour and under ambient room temperature. Friction coefficient was continuously measured during each test. At the conclusion of the test wear was measured in the test specimens by optical profilometry technique. Worn surfaces were also characterized by both optical and SEM microscopy to assess wear mechanisms.

The variation of fiction coefficient with time during dry unidirectional sliding test for various 4140 flat specimens is shown in Figure 28. After the initial run-in period of about 600 seconds, the friction coefficient for all the tested pairs became nearly constant for the remaining duration of the test. The magnitude of friction coefficient ranged from about 0.45 to about 0.85. A summary of the average friction coefficient after the run-in for the various flat specimens is shown in Figure 29. A variant of the FSP (1-12) has the lowest average friction of all the tested material, while another variant (1-14) has the highest friction. Although details of the reasons and mechanisms for the vastly different behavior in FSP treatment materials is not understood at this time, the observation suggests that FSP can be optimized for different frictional applications especially under dry sliding contacts.

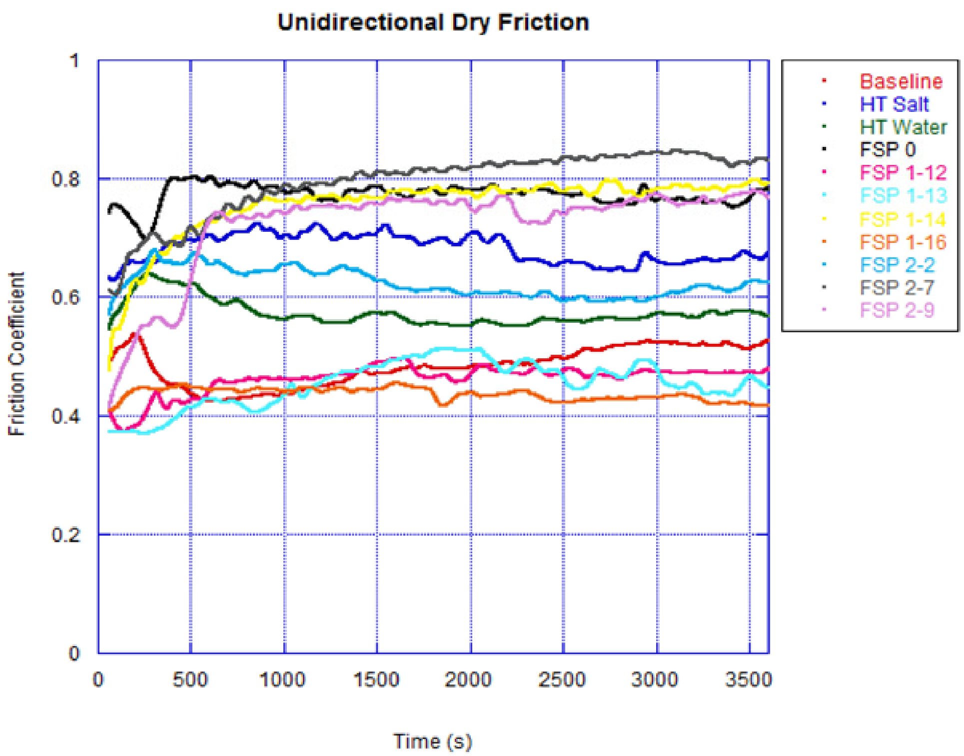


Figure 28: Variation of friction coefficient with time during unidirectional dry sliding

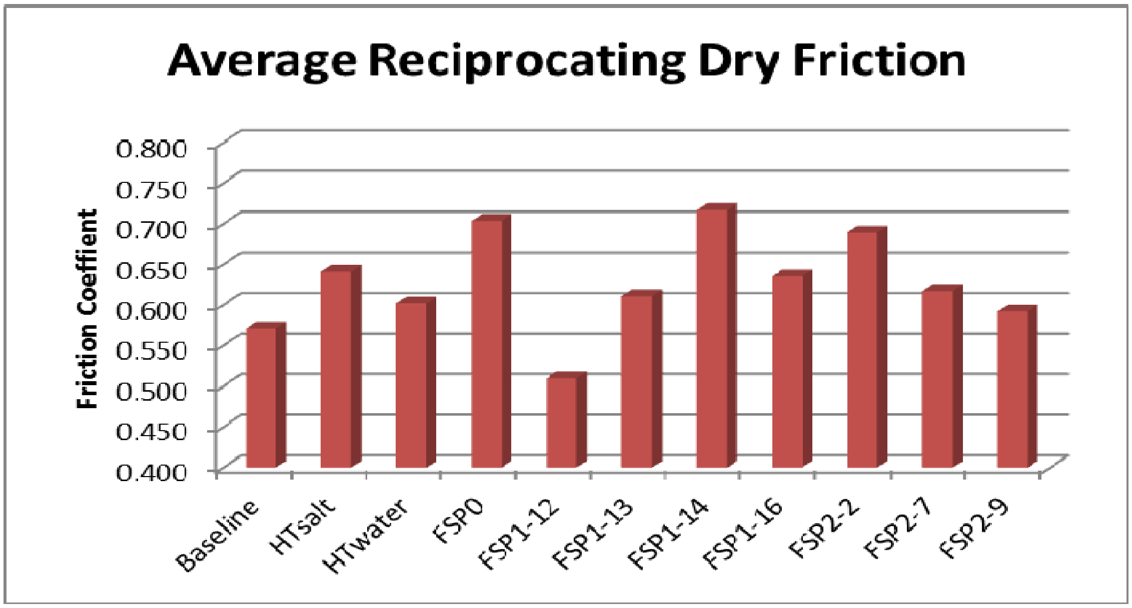


Figure 29: Average friction Coefficient during dry unidirectional sliding of different flats

The wear in various flat at the conclusion of the test is shown in Figure 30. All the surface hardening processes (heat treat and FSP) reduced wear compared to the baseline. However, significant differences can be seen in the wear of different variants of FSP. This observation again suggests that just as in friction, there is opportunity to optimize FSP processing for wear control. Indeed, more work is needed to develop adequate understanding and the role of FSP processing parameter on friction and wear behavior, especially under dry sliding contact.

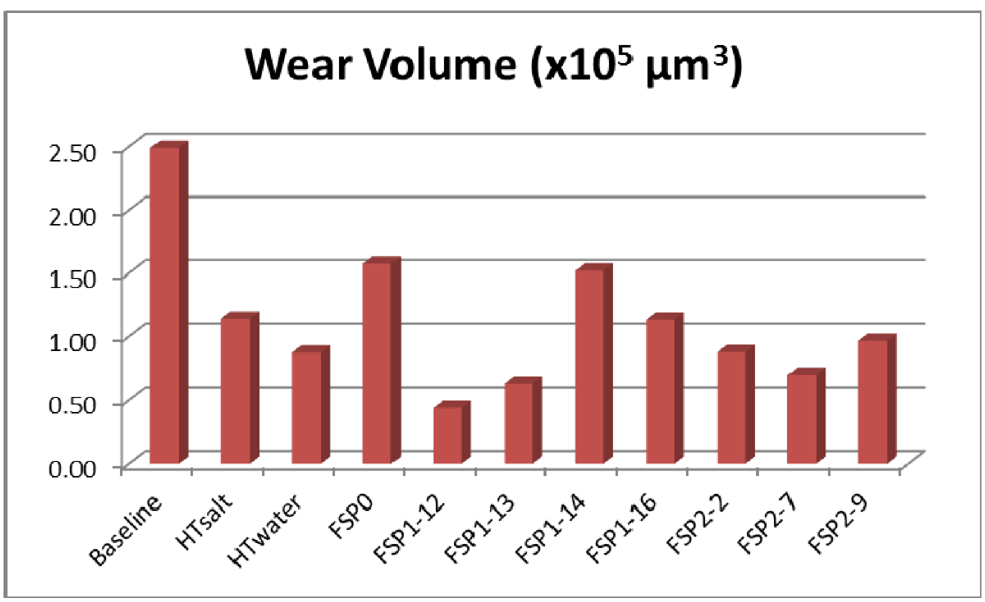


Figure 30: Wear Volume in flat specimens after dry unidirectional sliding test

Unidirectional sliding tests were also conducted under dry and lubricated conditions at a normal load of 5 N for the multi-pass FSP. Again, details of the test procedure were provided above.

A slight increase was observed in the unidirectional dry sliding for multi-pass treatment compared to single pass (Figure 31a). However, under lubricated condition, the friction for single pass and multi-passes are about the same; about 0.11 -0.13, which is typical for boundary lubrication regime.

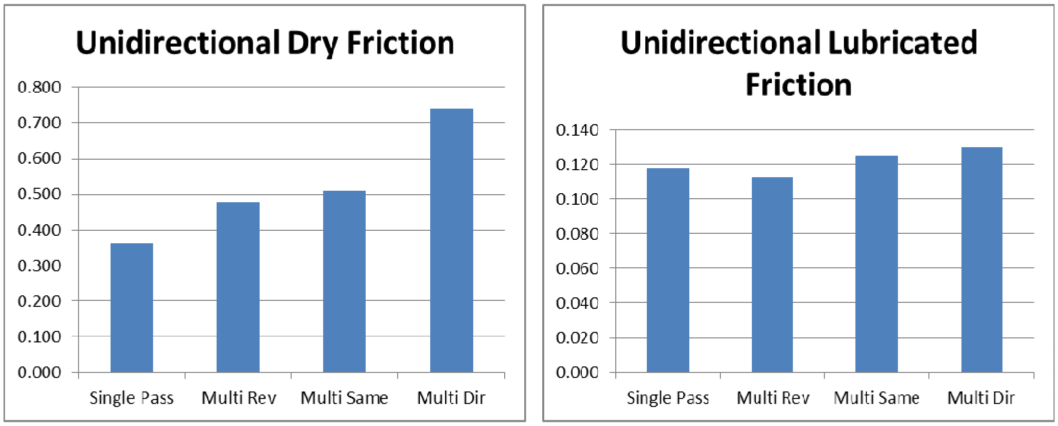


Figure 31: Average friction in unidirectional contact: a) dry and b) lubricated

Under dry condition, wear in multi-pass and single pass treatment are about the same (Figure 32a), while wear is reduced in the multi-pass treatment under lubricated condition when compared to single pass treatment (Figure 32b). These results again show that more work is needed to better understand the impact of number of FSP passes on friction and wear behavior.

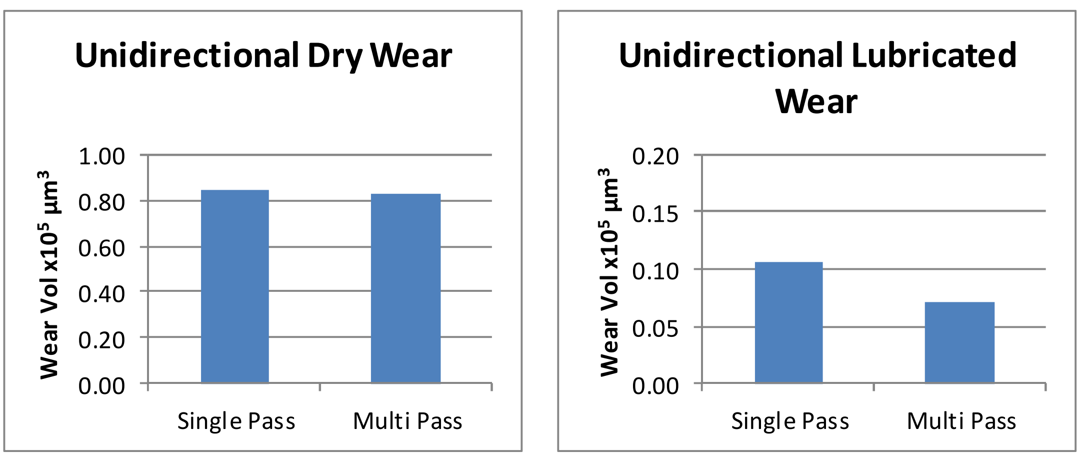


Figure 32: Average wear in unidirectional contact: a) dry and b) lubricated

Variation of hardness with number of passes in multi-pass treatment may provide some valuable insight. Figure 33 shows the variation of hardness as a function of depth from the FSP treated surface for different number of passes. There is a decrease in the case hardness for subsequent passes for the first three passes until a steady value is attained. This hardness behavior can be explained by the tempering action of subsequent passes until tempering is completed. This hardness behavior may in part, explain some of the friction and wear behavior.

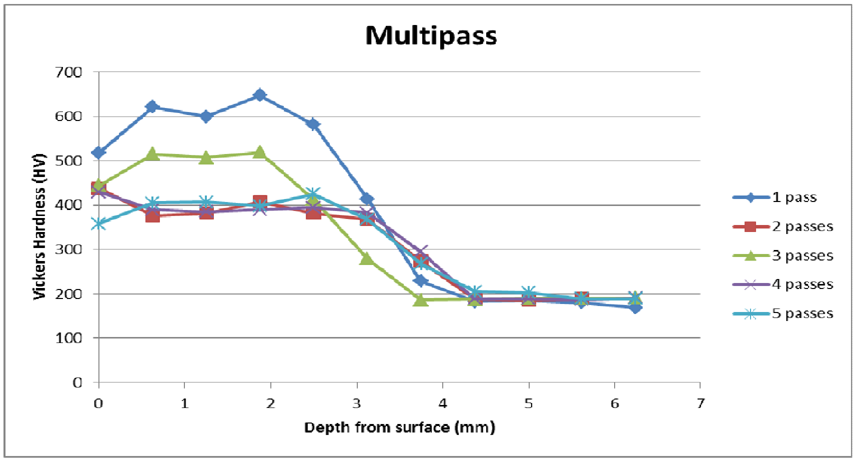


Figure 33: Hardness Profile

5.4 Task 4.0 Project Management and Reporting

All sub-tasks within this task were completed as planned, including

- Quarterly internal project team meetings
- Quarterly and final reports
- Project meeting with DOE Program officer
- Presenting initial results at a FSP conference and at a tribological conference.

6. Commercialization

Friction Stir Link's core mission is commercialization of friction stir welding and related processes. FSL's key technical team members have a history of development and implementation / commercialization of new technologies, even back to their previous employer in the automotive industry. Since the inception of the Friction Stir Link, major efforts and investment have been made in efforts to commercialize the friction stir welding technology. At the company's inception in 2001, there was virtually no friction stir welding or related production work being performed in North America. At that time, friction stir welding was mostly a laboratory level interest. After in excess of $5M of investment in equipment and related infrastructure, Friction Stir Link is the largest friction stir welding centric organization in the world. FSL has commercialized FSW in some of the most complicated applications to-date and currently performed approximately 500 hundreds miles of friction stir welding per year.

All of the data developed and ascertained from this concept project, would indicate that the state of development and the applications revealed, have the characteristics of other projects at Friction Stir Link that have shown a high rate of successful commercialization. While further development is certainly necessary, FSL believes the technology being developed and applied in this concept project has a high likelihood of being successfully commercialized.

7. Conclusions

The following conclusions have resulted from the investigation of FSP as an energy efficient alternative to traditional heat treatment or other surface material property modification processes.

- FSP is much more energy efficient than traditional heat treatment processes, with in excess of 95% energy savings possible. The energy savings primarily results from the fact that FSP is a local material property modification process, where traditional processes tend modify the entire part or surface.
- FSP cost is primarily driven by FSP tool cost and associated tool wear rates. FSP will have increased cost effectiveness or benefits versus traditional approaches, especially in applications where only a small amount of the surface needs to have modified material properties.
- FSP can increase surface hardness to a level equal to or greater than traditional heat treatment processes.
- FSP can improve friction and wear properties to a level equal or greater than traditional heat treatment processes.
- The effect of FSP on tribological material properties is variable versus FSP parameters and FSP tool design. This indicates further optimization is possible.
- Results from this project indicate that FSP of steel can be a viable production process.

8. Recommendations

The following recommendations have resulted from the investigation of FSP as an energy efficient alternative to traditional heat treatment or other surface material property modification processes.

- In this project, applicability of FSP was only investigated towards steel alloy 4140. There are other steel alloys that are used in parts with relative motion. It is recommended that studies be performed with FSP of other alloys.
- With FSP requiring high processing forces and a number of potential applications have relatively limited access, studies need to be performed into reducing FSP forces and miniaturization of FSP equipment, so as to expand the base of potential application of FSP where enhanced tribological properties are of benefit.
- Beyond heat treatment, there are multiple processes by which the near surface material properties can be further enhanced by placing the components in a non-air atmosphere environment. Studies of FSP in alternative non-atmospheric environments should be performed to determine if further enhancements or improvements can be realized with the use of FSP.
- In view of the significant observation of the effect of FSP processing variables and parameters on the friction and wear behavior of steel material, a better understanding of the mechanisms by which FSP enhances friction and wear behavior of materials is needed. Such knowledge is essential for effective and efficient deployment of FSP technology for tribological applications.

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10. Appendix

Equations for Energy usage and cost:

FSP Cost Equations:

Total Distance of FSP on Circumferential Area

$$d = \pi D w / \phi$$

d = Total Distance of FSP
D = Diameter of Part
w = Width of Part
ϕ = Diameter of FSP tool or Width per FSP pass

Cost per Part

$C_f = C_t + C_m$

C_f = Total Cost per Part
C_t = Cost related to FSP Tool per Part
C_m = Cost related to FSW Machine use and Labor Part

$C_t = F_t D/L$

F_t = Friction Stir Tool Cost
L = Tool Life

$C_m = M_f D/T_w$

M_f = FSP Machine Charge Rate
T_w = FSP Travel Speed

Heat Treatment Cost Equations:

$C_h = M_h T_h / n$

M_h = Heat Treatment Machine Charge Rate
T_w = Total Time to perform Heat Treatment Process
n = Number of parts in heat treatment oven

FSW Energy Calculations

FSP Energy per Part

$E_f = \tau\, \omega\, d / T_w$

τ = Measured FSP
ω = FSP Rotation Speed
d = Distance of FSP
T_w = Travel speed of FSP

Heat Treatment Energy Per Part

Data provided by heat treatment suppliers in British Thermal Units of Gas used per batch and converted to kilo-watt hours for comparison to FSP energy.