H. Friction Stir Joining and Processing of Advanced Materials Including MMCs

Principal Investigator: Glenn J. Grant Pacific Northwest National Laboratory

P.O. Box 999, 902 Battelle Blvd., Richland, WA 99352

(509) 375-6890; fax: (509)375-44; e-mail: glenn.grant@pnl.gov

Project Manager: Moe Khaleel Pacific Northwest National Laboratory

P.O. Box 999, 902 Battelle Blvd., Richland, WA 99352

(509) 375-2438; fax: (509) 375-6631; e-mail: moe.khaleel@pnl.gov

Chief Scientist: James J. Eberhardt

(202) 586-9837; fax: (202) 587-2476; e-mail: James.Eberhardt@ee.doe.gov

Field Technical Manager: Philip S. Sklad

(865) 574-5069; fax:(865) 576-4963; e-mail: skladps@ornl.gov

Participants:

Bill Arbegast, Glen Stone, Stan Howard, Casey Allen Advanced Materials Processing Center South Dakota School of Mines and Technology Rapid City, S.D.

Contractor: Pacific Northwest National Laboratory

Contract No.: DE-AC06-76RL01830

Objective

• Develop Friction Stir Joining and Processing (FSJ / FSP) to enable more widespread use of lightweight, advanced materials in heavy vehicle manufacturing.

Approach

- Develop new technologies within the broad topic area of friction stir joining and processing that facilitate lightweight manufacturing methods and new materials for use in heavy vehicle application.
- FY 2005 the project focused on three primary areas:
 - Use FSP to create engineered surfaces on lightweight materials that display enhanced surface properties
 - Use FSJ to join sheet materials for superplastic forming applications
 - Develop fundamental process models using a new approach: Smooth Particle Hydrodynamics (SPH)

Accomplishments

- Developed methods to incorporate ceramic powder into the surface of aluminum and cast iron to produce engineered surfaces for improved wear resistance.
- Developed the new area of Friction Stir Reaction Processing to create engineered surfaces that contain solid state, in-situ formed phases including nano-sized particles that impart unique properties to a material (increased strength, hardness, ductility, and fatigue life).
- Developed FSJ weld metal with superplastic properties to facilitate large multi-sheet SPF or integrally stiffened
 panel applications for heavy vehicle cab structures.

 Developed unique computational code based on SPH to predict material flow, mixing and defect formation in FSJ/P.

Future Direction

- Initiate wear testing of engineered surfaces created using FSP.
- Develop weld process parameters that enhance superplasticity in joined assemblies to allow for part consolidation in light-weight SPF cab structures.
- Test multi-sheet and integrally stiffened panel for strength, modulus and other mechanical performance metrics.
- Parallelize SPH computational code to run on supercomputers to improve the ability of the model to define fine features of flow, defect formation and tool designs in friction stir joined or processed material.

Introduction

One of the key strategies for making a vehicle energy-efficient is to manufacture it from lighter materials. Structural and functional requirements, however, lead to a situation where no single lightweight material is appropriate for all applications. A modern, weight-optimized vehicle structure is a hybrid of many materials. A critical problem that has emerged in the development of these hybrid structures is that for many material combinations, traditional joining technologies (e.g., fusion welding or mechanical fastening) are not appropriate. For some highly specialized materials, like aluminum MMCs, titanium, and advanced high-strength steels, a better joining technology can have significant impact on whether these materials have a role in future vehicle structures.

In the last 15 years, a new joining technology, Friction Stir Joining (FSJ) has emerged and has the potential to join many lightweight materials. Invented by TWI, Ltd., FSJ is a solid state process that employs severe plastic deformation to create joints between a wide variety of different materials. A typical FSJ butt joint is depicted in Figure 1. The weld is created by clamping the materials to be joined and plunging a spinning tool into the surface. The spinning tool is then translated down the joint line, leaving behind a weld zone characterized by a fine-grained, dynamically re-crystallized microstructure. Typically, the tool is spun at 400 rpm to 2000 rpm and translated down the joint line at a rate of 4 to 300 in./min, depending on tool design, base material, and thickness. As the tool rotates and translates, complex flow patterns

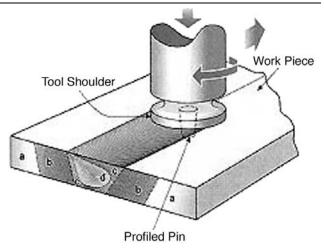


Figure 1. Friction stir joining and processing are accomplished by plunging a spinning tool into a material and translating the tool across the surface to form either a joint or a surface processed region (TWI, Ltd.).

develop in the base material that creates an intimate mixing of materials from both sides of the weld. Heat input during plastic deformation generally creates a temperature in the weld between 0.6 and 0.8 of the absolute melting temperature so that no liquid phase is generated.

FSJ is capable of producing aluminum and magnesium alloy welds as good as or better than fusion welds in terms of joint efficiency, mechanical properties, and environmental robustness. A significant advantage of the process, for application to hybrid structures, is that since there is no melting during the process, a large variety of dissimilar material joints are possible, including dissimilar aluminum and magnesium joints that are not possible with conventional fusion welding. In the last five years, FSJ has been shown to be a commercially important, energy-efficient, and

environmentally friendly process for joining aluminum. However, there are many opportunities for other higher-strength lightweight materials to be considered if good joining technologies for these materials existed as well. The objective of this project is to investigate how FSJ can be applied to advanced materials including AL-MMCs, titanium, steels, cast iron, and materials that display graded structures with unique surface properties. Moving the FSJ process from "soft" materials like aluminum and magnesium into advanced, higher-strength alloys has proved to be challenging because of the mechanical and thermal demands on the tool materials. In steels, for instance, the tools must survive high forge loads as well as tool temperatures of up to 1100°C.

Friction Stir Processing

Recently, a new research direction has emerged as an outgrowth of FSJ that recognizes that the same solid state deformation process can be used to modify the surface of a monolithic material for enhanced properties. This new research direction is called Friction Stir Processing (FSP).

During previous years, this project demonstrated that it is possible to create a particle-reinforced zone of 20-micron SiC or Al₂O₃ particles in a 6061 aluminum base alloy (Figure 2). Microscopy has shown that the stirred region is developed as deep as the pin probe (up to 0.25 inches in our tests), it is defect-free and it forms a graded metallurgical bond with the underlying surface. No interface is developed between the composite zone and the base material. The surface zone has the potential to be orders of magnitude thicker than conventional coating technologies; and it has the added benefit of producing a graded structure that does not have a sharp interface with the underlying substrate, thereby avoiding many of the problems seen in conventional coatings (coefficient of thermal expansion mismatch, etc.).

While lightweight wear-resistant materials could benefit from this compositing technique, perhaps the greater application could be in ferrous or hard alloy systems. Hard particle reinforcement of the surfaces of steels, titanium, brasses, or cast iron may have

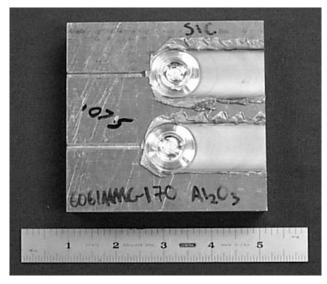


Figure 2. Friction stir processed 6061 plate with ceramic particulate incorporated into the surface.

numerous industrial applications in reciprocating assemblies, engines, brake disks, or other situations where both bulk strength and surface wear resistance is needed. Lightweight high-strength steels with surface wear resistance may have numerous applications in both lightweight structures and lightweight vehicle power systems. FSP work during FY 2005 focused on developing methodologies to successfully surface-process high-temperature materials including steels and cast iron.

Approach

The basic objective of this project is to investigate and develop FSJ and FSP as viable industrial techniques for advanced lightweight materials. The current approach is divided into three main task areas: (1) explore the potential to modify the surfaces of both conventional aluminum and advanced materials toward the goal of improving wear, corrosion, mechanical or thermal properties, (2) develop FSJ/FSP as a process to join sheets that will be subjected to superplastic forming, and (3), to develop numerical, physics-based modeling strategies to help define the fundamental process and predict process conditions.

The first task focused on broadly investigating using FSP to create engineered surfaces. Surface modification was investigated in two areas:
(a) particle incorporation to create near-surface MMCs in cast iron, and (b) surface alloying by

reaction processing. In addition, to facilitate FSP in cast iron, we investigated the use of induction preheating to lower process forces in these hard-to-process materials.

The second major task area investigated in FY05 was the application of FSJ to superplastic forming environments. Numerous opportunities exist to use this manufacturing technology to reduce weight on heavy vehicles. One barrier to SPF manufacturing is that hang-on truck components are complex and often very large; larger than the standard coil widths. If sheets are joined together to make a large part by fusion welding, the weld region does not deform superplastically because of the microstructure of fusion weld metal. Friction Stir Joining produces a weld region that is formable by SPF and may be an enabler for complex or large structures that are joined, then later hot gas formed in single operations to form cab structures and panels.

The third major task area was the development of a numerical modeling process using an approach called Smooth Particle Hydrodynamics (SPH). This modeling approach has not been previously applied to FSJ/P but our preliminary work suggests that it may be able to provide significant insight into the fundamental nature of the heat generation and material flow that occurs during the FSJ/P process.

Results

Task 1 Surface Engineering – Cast Iron/TiB₂ FSP

The motivation for looking at surface engineering of the cast iron system comes from the need in many industrial environments to produce a more wear resistant cast iron. The bulk of the work in FY2005 on the FSP of cast iron has concentrated on developing methods to stir-in ceramic particulate into the surface of cast iron. This can produce a wear resistant surface on a material while retaining the original bulk properties. Cast iron is a difficult material to FSP and much of the FY05 effort was devoted to developing the process parameters and tool designs and materials needed to successfully process this material.

Two main tool designs and two tool materials were investigated during the program. The two tool designs used are the Stepped Spiral design and the

Three Flat tool shown below in Figure 3. The two tool materials investigated were commercial W-25% Re and Polycrystalline Cubic Boron Nitride (PCBN).

Wear of the pin tool was found to be a significant problem initially requiring several strategies to be employed to mitigate the wear. Most important of these is the use of induction preheating of the substrate cast iron prior to tool traverse. Research at the South Dakota School of Mines and Technology has shown that induction preheating of the substrate prior to FSP can have an important effect on process forces and perhaps on the quality of the weld in difficult-to-weld materials. Figure 4 shows the

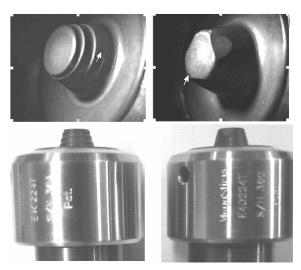


Figure 3. Stepped Spiral tools and Three Flat tools were fabricated in W-25%Re, and Stepped Spiral tools were fabricated in PCBN for use in the program.

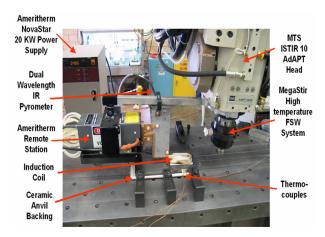


Figure 4. Induction Preheater installed on FSJ equipment at the South Dakota School of Mines and Technology

friction stir machine. Figure 5 shows that for 1018 steel, induction preheating can reduce process forces throughout the weld or processed region length [Ref 1]. Reduced process forces can be correlated with lower tool wear and better material flow in the weld. Induction preheating was found to be a necessary procedure in the FSP of cast irons to help reduce tool wear. It was found however that even with induction preheating, tool wear was still a significant problem with the W-25%Re tools. PCBN tools were found to be more wear resistant in this material system. In addition, the Stepped Spiral tools produced better nugget microstructures and larger plasticized zones in testing than the Three Flat tool designs. Figure 6 illustrates the current experimental setup found to promote the best tool survival and produce the best nugget microstructures. It was found that the initial plunge of a cool tool into a cool cast iron substrate also created significant wear. To mitigate this, a thin sheet (0.080" thick) of 1018 steel was placed on top of the cast iron. This plate of less abrasive material plasticizes first and allows heat to develop in the cast iron substrate, lowering the flow stress and producing a less aggressive wear environment for the pin during the plunge phase. The plate is also important for lowering x axis forces during processing, also decreasing pin tool wear.

Figure 7 shows nugget microstructure in cast iron and is typical of FSL/P in many material systems. Figure 8 shows a microhardness traverse across the nugget of the friction stir processed cast iron.

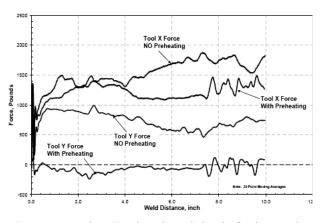
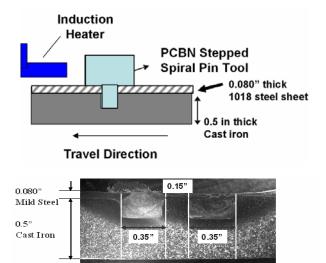


Figure 5. Work at South Dakota School of Mines and Technology has shown that induction preheating can lower process loads in difficult-to-weld materials like 1018 steel. (Figure courtesy of Tweedy, Arbegast, Allen, 2004).

Hardness is elevated well above the parent material in the HAZ, TMAZ and in the Nugget. Detailed microstructural investigation (Fig. 9) show the nugget is composed of fine grain ferrite and pearlite with all the coarse graphite dissolved in the matrix.

Experiments were also conducted in which 10 to 20 micron TiB₂ powders were introduced between



Picture of the weld of cast iron with mild steel on the top



Picture of the weld ground to the required thickness

Figure 6. Top figure shows experimental set up. Middle figure shows as-processed cast iron with .080 plate on surface. Lower figure shows FSP cast iron with steel plate machined off, exposing nugget to wear surface. Final demo plates will be friction stir processed so that nuggets overlap.

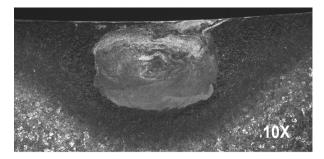


Figure 7. Cast Iron nugget microstructure showing consolidated nugget region.

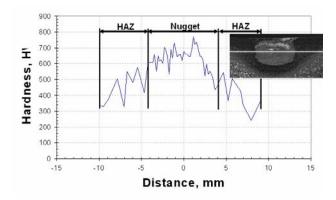


Figure 8. Hardness across FSP zone showing increased hardness in HAZ and Nugget over base material.

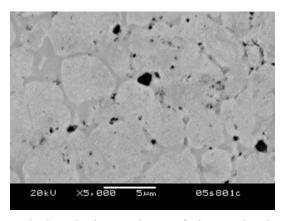


Figure 9. SEM backscatter image of stir zone showing 50 to 200 nm TiB2 in a fine grained ferrite / pearlite matrix (Uma Ramasubramanian, et. al. 2005)

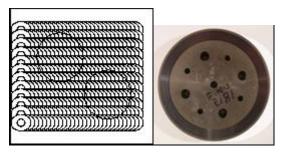


Figure 10. Overlapping pattern of FSP "beads" laid down on cast iron plate, then 4 inch diameter circular plates cut out and tested for wear performance

the 1018 steel plate and the cast iron substrate then the friction stir tool was passed through the assembly. These ceramic powders were successfully mixed into the nugget region to create a homogeneous distribution of ultrafine grained TiB_2 (50-200 nm). It is interesting to note that some of the fine TiB_2 powders reacted with the carbon in the cast

iron to form TiC particles. This reaction occurs in the solid state and may enhance the wear properties of these surface engineered regions.

Friction Stir Reaction Processing (FSRP)

Much of the effort in FY 2005 was directed toward the new field of FSRP. Thermodynamic calculation of numerous potential solid state reactions indicates that energies available during FSJ/P may be high enough to initiate the formation of some compounds like TiB₂ from elemental constituents, especially considering all the new surface being generated by the severe plastic deformation under the pin tool. This process opens the possibility of making very finely divided reaction products in the near-surface region of a bulk material (see Table 1). These reactions can be tailored by the composition of the elemental powders introduced on the surface. In addition, highly exothermic (thermite style) reactions may be possible that can put large amounts of heat into the surface and potentially reduce the flow stress of hard-to-weld alloys, or allow the FSJ process to occur without significant tool wear. Claddings of materials rich in fine oxide dispersions. or other in situ-formed ceramic-rich materials, may be possible on low-cost ferritic base alloys (creepresistant surfaces for engine applications). Work in FY 2005 concentrated on reactions in the Mode I category in Table I. These are metal oxides reacting with the aluminum substrate to form alumina and free metal.

Reactant powders were placed between plates of 1100 alloy aluminum as shown in Figure 11. The assembly was then friction-stir-processed to produce a microstructure, as shown in Figure 12. The nugget region contains very finely dispersed precursor reactants and SEM EDX reveals the presence of reduced metal phases, indicating that in-situ reaction did take place during the FSP.

Experiments were conducted with powders composed of SiO2, TiO2, CuO, NiO, in aluminum and NiO in steel. All experiments showed development of fine reduced metal or silicides and aluminides indicating chemical/mechanical assisted reactions are taking place. Interestingly the alumina phase that is the oxide reaction product was not identified in SEM suggesting that it may be an ultra fine grained, potentially nano-phase. More work in

Table 1. Reaction modes

Mode	Reaction	Examples
I	Base Metal (Al) + Reactant	$3 \operatorname{SiO}_2 + 4\operatorname{Al} \longrightarrow 3\operatorname{Si} + 2\operatorname{Al}_2\operatorname{O}_3$
	Product	$3 \text{ TiO}_2 + 4 \text{Al} \longrightarrow 3 \text{Ti} + 2 \text{Al}_2 \text{O}_3$
		$BN + Al$ \longrightarrow $AlN + B$
II	Pin Tool (Ti) + Reactant	$3 \text{ Ti} + 2 \text{ BN} \longrightarrow 2 \text{ TiN} + \text{TiB}_2$
	Product	$Ti + AlN \longrightarrow TiN + Al$
		$Ti + C \longrightarrow TiC$
		$Ti + 2B \longrightarrow TiB_2$
III	$A + B \longrightarrow C + D$	$4Al_{(powder)} + 3SiO_2 \longrightarrow 3Si + 2Al_2O_3$
		$4Al_{(powder)} + 3TiO_2 \longrightarrow 3Ti + Al_2O_3$

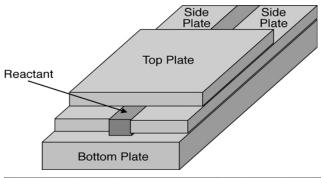


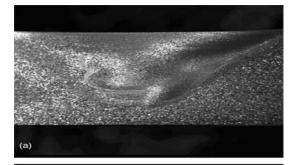


Figure 11. Reactant oxide powders are placed between plates, and the stir tool processes the entire thickness

2006 is planned to see if this process might be able to create surface regions enriched in ultra fine grained alumina. If so, unique properties in the surface layer may be possible, such as ultra hardness or creep resistance as is seen in other nanophase alumina dispersions.

Task 2 - FSJ/P for SPF manufacturing

Superplastic forming (SPF) is a lightweight manufacturing technology that has shown to be cost effective and produce weight optimized structures



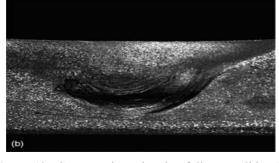


Figure 12. Cross sections showing fully consolidated weld zone. FSRP zone is 3/8-in (9.52 mm) thick: (a) SiO₂ and (b) TiO₂.

through reduction in part count and through the use of lightweight materials.

One important factor that is relevant to the particular problems of heavy vehicle SPF is that truck parts are large. The parts are modulus (stiffness) driven, not strength driven, because of aero flutter and shear size. Big parts need stiffeners. This requires a complex assembly. One cost effective and lightweight option is to join multiple flat sheets together, then SPF the entire multi-sheet pack together in one operation. This incorporates the

stiffener into the assembly. The problem with this strategy revolves around the method chosen to join the sheets together. If the sheets are fusion welded, the microstructure of the fusion weld region will not deform superplastically. Figure 13 shows the coarse microstructure of a fusion welded aluminum sheet. When these joints are subjected to superplastic forming the joint remains un-deformed. These undeformed areas also have very poor post-forming mechanical properties. In contrast, Friction Stir Joining leaves behind a weld microstructure that is highly deformable at SPF conditions.

This project is investigating the particular weld process parameters and part geometries that could produce an integrally stiffened panel for a heavy vehicle cab application. The work to date involves fabricating multi-sheet assemblies as shown in Figure 14. These assemblies will be SPF formed into structural panels in three configurations, "egg crate", "hat stiffened", and "doughnut". The purpose of the test forms is to discover the correct FSJ process parameters and the SPF deformation possible in these geometries. The panels are made using both linear FSJ and Friction Stir Spot Welding (FSSW) (Fig 15, 16). The FSSW joints are of particular

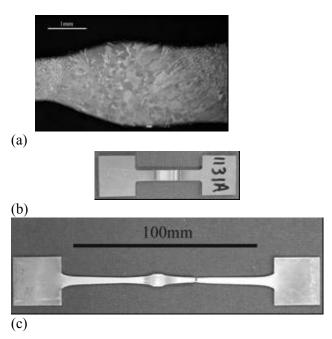


Figure 13. (a) Weld nugget region in a butt weld between aluminum sheet materials showing coarse, cast microstructure of a fusion weld. (b)Transverse tensile specimen (c) Transverse tensile test at SPF conditions shows the undeformed nature of the fusion weld metal.

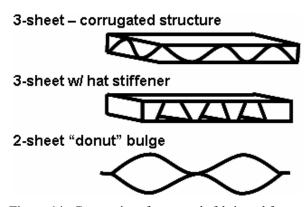


Figure 14. Geometries of test panels fabricated for this program

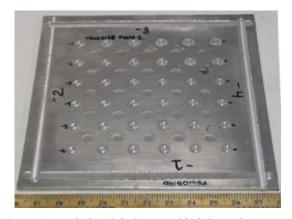


Figure 15. Friction Stir Spot Welded three sheet multisheet pack prior to SPF testing. This configuration is designed to produce the corrugated structure. Alternating spot welds are made from each side of the multisheet pack using the "Refill" (GKSS-Riftec) FSSW method at the SDSMT.

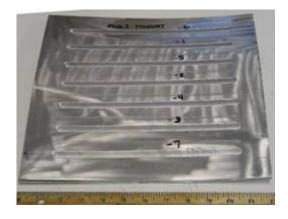


Figure 16. Linear Friction Stir Welded multisheet pack prior to SPF testing. This configuration is designed to produce the "3 sheet with hat stiffener" structure. Alternating linear welds are made from each side of the multisheet pack using a pin that penetrates only through the upper two sheets.

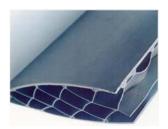
interest because they are made using a new process that does not leave an exit hole after the spot joint is made. This process, called the "refill" method was invented by GKSS (Riftec) and the panels were fabricated at the facilities of our project partner, South Dakota School of Mines and Technology.

It is envisioned that structural panels like those in Figure 17 can play a key role in a vehicle light weighting program. The panel shown in Figure 17 is fabricated in titanium. To fabricate the same structure in aluminum will present a significant challenge because of the aluminum oxide surface, and because of a particular feature of highly worked aluminum alloys that are subject to high temperature called Abnormal Grain Growth (AGG). Abnormal Grain Growth can limit the SPF forming performance and seriously restricts the post forming mechanical properties. AGG can be minimized with the correct choice of FSJ process parameters, but whether it will be a significant barrier to implementation has yet to be determined.

Task 3 - Numerical Modeling

Work also began in FY2005 on a new modeling approach to investigate the fundamental flow and heat generation properties of friction stir welds. The industry has struggled with modeling this process for 10 years because the high strains and high strain rates are simply not seen in very many other systems. Conventional modeling techniques such as Finite Element Modeling and Computational Fluid Dynamics suffer from many issues mostly surrounding the need for the model to track huge variations in strain, temperature-dependent flow stress, strain rate, and heat generation.

The modeling approach we have started to investigate in this program is a departure from other



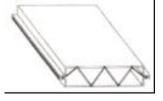


Figure 17. Possible structures formed from multisheet packs joined together as sheets then subsequently hot gas formed into a lightweight structural panel

workers in the field and involves the use of Smoothed Particle Hydrodynamics. This is a fully Lagrangian particle method, so no grids are required. The code solves 2D and 3D equations for continuity, momentum, and energy balance. Currently the momentum equation is based on a fluid dynamic (Navier-Stokes) equation, but solid mechanics equations are being adapted so the materials databases are easier to incorporate as input parameters.

The model currently can explicitly model mixing of materials, predict temperature generation at the surface of the tool as well as that due to viscous dissipation and can produce temperature and strain histories for any material point in the field. This may allow it to make micro-structural prediction at different parts of the weld since temperature and strain history can be tracked, although this has not as yet been implemented in the code. Figure 18 shows some example output from the model.

Conclusions

FSJ and FSP are technologies that will enable the application of many lightweight materials in the next generation of transportation systems. Many advanced materials are in need of effective joining technologies before their widespread use can be considered. Solid state FSJ/P avoids many of the problems with fusion joining and represents a revolutionary change in joining technology. FSP also has numerous opportunities in the growing field of surface engineering and thermal management.

The results of this work will allow designers to anticipate and implement structures that are a hybrid of many different materials, facilitate the application of lightweight superplastically formed structures, and suggest new materials and engineered surfaces that can help deliver lighter and more fuel-efficient vehicles.

Acknowledgements

The authors would like to acknowledge the substantial support over the course of this multi-year project given by Dr. Sidney Diamond. Dr. Diamond carried a high level of enthusiasm and a vision for how new technologies, such as friction stir processing, might be inserted into the heavy vehicle

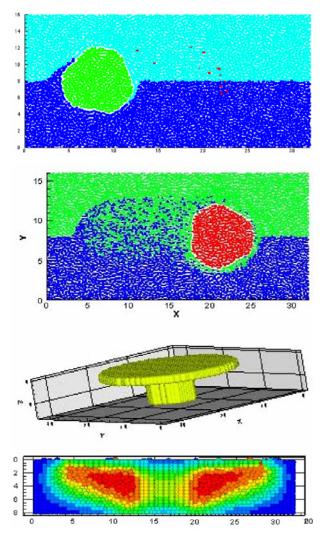


Figure 18. Model output. Upper two figures show mixing of dissimilar materials using a tool with three flats on the pin. (Only the tool pin is shown in this 2D simulation. The lower two figures show arrangement of 3D model and a temperature distribution. Highest temperatures are in a region near the pin root away from the pin surface, due to heat generated by plastic work and conduction into the pin.

manufacturing industry to help accomplish the goals of the Department of Energy's Freedom Car and 21st Century Truck programs. His guidance is appreciated.

References

 B. M. Tweedy, W. Arbegast, and C. Allen, "Friction Stir Welding of Ferrous Alloys using Induction Preheating," in *Friction Stir Welding* and Processing III, ed. K. V. Jata, M. W. Mahoney, and T. J. Lienhert, TMS, 2005.

Presentations/Publications

- G. J. Grant, *FY 2004 Annual Report:* Friction Stir Joining and Processing of Advanced Materials, Including Metal Matrix Composites, Nov 2004.
- G. J. Grant, *PNNL Milestone Report* PNNL 21346 Evaluation of friction stir processing of steel for thermal barrier coating. Feb 2005
- S. M. Howard, W. Arbegast, B. Jasthi, G. J. Grant, D. R. Herling, "Friction Surface Reaction Processing On Aluminum Substrates,"," in *Friction Stir Welding and Processing III*, ed. K. V. Jata, M. W. Mahoney, and T J. Lienhert, TMS, 2005
- U. Ramasubramanian, W. Arbegast, G. Stone, G. J. Grant, "Friction Stir Processing of Class 40 Cast Iron," in *Friction Stir Welding and Processing III*, ed. K. V. Jata, M. W. Mahoney, and T J. Lienhert, TMS, 2005