

## **E. Friction Stir Joining and Processing of Advanced Materials Including Metal Matrix Composites**

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## **Objective**

- Investigate and develop friction stir joining (FSJ) and friction stir processing (FSP) as viable industrial joining and processing techniques for advanced materials, including aluminum metal matrix composites (MMCs), titanium, and advanced high-strength steel. During FY 2004, this project focused on two main task areas: the development of appropriate tooling and process parameters that will allow friction stir welding (FSW) to be used in abrasive and/or high-temperature materials, and the use of the FSP process to modify the surfaces of materials for advantageous near-surface mechanical and thermal properties.

## **Approach**

- Develop new tooling designs and fabricate tools from advanced tooling materials (Ni-alloy and Fe-matrix TiC MMCs).
- Develop weld process methods, such as induction preheating, to lower process forces and reduce wear or tool breakage issues in hard-to-weld materials.
- Experimentally determine the feasibility of making near-surface graded composites by FSP in aluminum and steel to create functionally graded materials with enhanced surface-engineered properties.
- Investigate the new method of friction surface reaction processing (FSRP) in which a friction stir tool is used to initiate in situ solid-state reactions that can stabilize new phases or ultra-fine grained phases in the near-surface regions of bulk materials for enhanced surface properties.

- Investigate the application of FSJ to ferritic oxygen-dispersion-strengthened (ODS) alloys, including MA957.
- Investigate the use of FSP to create robust thermal barrier coatings on steel.

### Accomplishments

- Fabricated tools using new unique designs that will be used in coatings and wear studies.
- Developed, at the South Dakota School of Mines and Technology, induction preheating techniques that facilitate FSJ/P in steels and cast iron.
- Created near-surface regions in aluminum and steel that are enriched in ceramic particulate by physically stirring powders into the surface using a spinning friction stir tool.
- Began experimental studies into FSRP by plasticizing and reacting elemental powders and oxides beneath a spinning tool during FSP.
- Performed preliminary experiments on using FSP to plasticize MA957, a ferritic ODS alloy.
- Used FSP to process pre-existing thermal barrier coatings on steel to create diffuse, interface-free, near-surface zones that have unique thermal properties.

### Future Direction

- Further develop tool materials and designs for steel and titanium FSJ/P
- Develop process parameters and conditions necessary for surface modifications of steel and titanium
- Develop friction stir surface processing for thermal barrier applications
- Develop the process of reaction surface processing to create, by solid-state chemical reaction, new phases at the surface.

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## Introduction

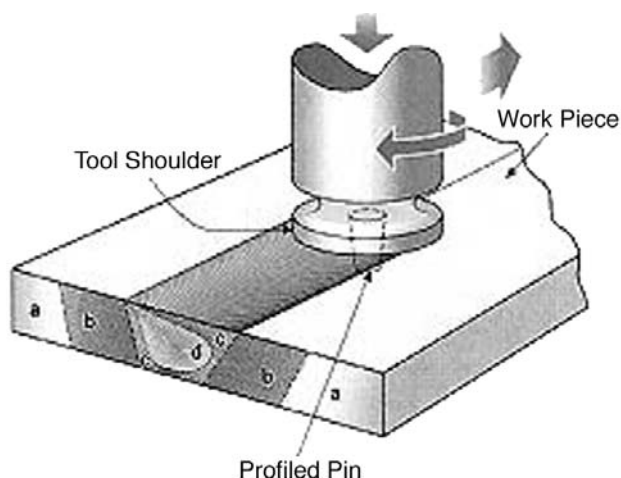
### Friction Stir Joining

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, the new joining technology FSJ has emerged that 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 recrystallized 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 develop in the base material that create 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



**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.).

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 there existed good joining technologies for these materials as well. The objective of this project is to investigate how FSJ can be applied to advanced materials including AL-MMCs, titanium, and steels. 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. A primary challenge is to develop pin tool designs and materials that can survive the high-temperature and/or abrasive-wear conditions under which the tool will be forced to operate.

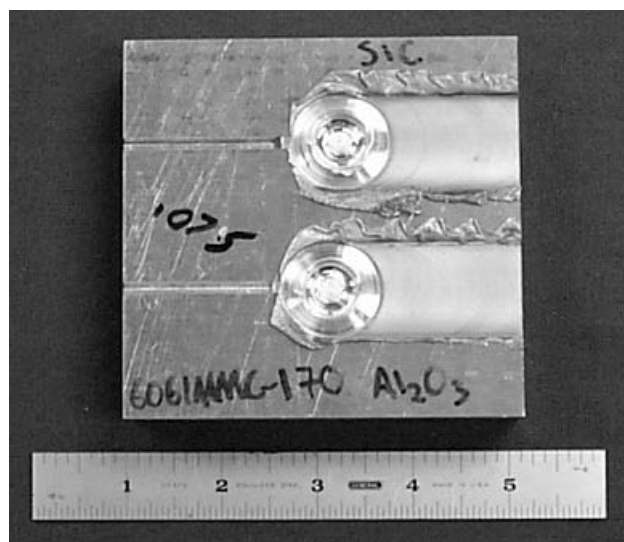
### **Friction Stir Processing**

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 mod-

ify the surface of a monolithic material for enhanced properties. This new research direction is called FSP.

Several applications of FSP have been investigated during the course of this project including surface modification for wear resistance, for creation of bulk superplastic properties, and for improvement to the near-surface defect and porosity distribution in Al-MMC castings. Work during FY 2004 concentrated on creating wear-resistant surfaces by FSP particle incorporation, on creating engineered surfaces by solid state reaction- processing, and on basic experiments in creating thermal barrier coatings by FSP.

This work was designed to test the feasibility of using FSP to create engineered surfaces. During previous years, this work demonstrated that it is possible to create a particle-reinforced zone of 20-micron SiC or Al<sub>2</sub>O<sub>3</sub> particles in a 6061 aluminum base alloy (Figure 2). Microscopy has shown that the stirred region is developed as deep as the pin probe (2–3 mm in our tests), which is defect-free and forms a graded metallurgical bond with the underlying surface. No interface is developed between



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

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 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. Work during FY 2004 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 approach used has been to (1) seek to develop new tool materials and designs that will allow successful joins to be made in Al-MMCs, titanium, and steel; and (2) explore the potential to modify the surfaces of both conventional aluminum and magnesium alloys and advanced materials toward the goal of improving wear, corrosion, or mechanical properties.

The FY 2004 program is divided into three main tasks. The first task focused on developing tool designs and fabricating tools in some of the tool materials defined by last year's efforts. The second task area broadly investigated using FSP to create engineered surfaces. Surface modification was investigated in three areas: (1) particle incorporation to create near-surface MMCs in aluminum and steel, (2) surface alloying by reaction processing, and (3) surface modification of pre-existing thermal barrier coatings to possibly create graded structures with unique thermal properties. In addition, to facilitate FSP in steels, we investigated the use of induction preheating to lower process forces in these hard-to-process materials. Finally, the third main task was a

small investigative effort into the feasibility of using FSJ to join or process a ferritic ODS alloy, MA957. This material is a creep-resistant high-temperature steel that has potential application in high-temperature environments such as solid oxide fuel cells.

## **Results**

### **Tool Materials and Designs**

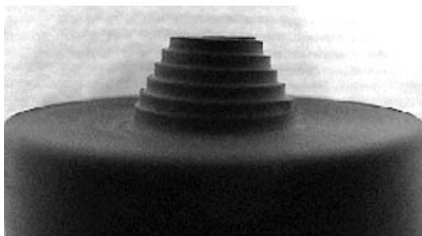
During FY 2003, promising tool materials were identified for each material system. In FY 2004, tools were fabricated from some of these materials, and the tools were used to process aluminum and steel surfaces. Figure 3 shows several tools made from H13 (baseline), MP159 (a cobalt-nickel alloy), and FerroTiC (an iron-based TiC MMC). These tool materials show virtually no wear during FSJ of standard aluminum alloys. However, creating surface-modified regions enriched in ceramic particles requires tool materials to have strong wear resistance. In aluminum MMC systems with either SiC or Al<sub>2</sub>O<sub>3</sub> particulate, H13 and nickel alloy materials showed significant wear, while FerroTiC showed



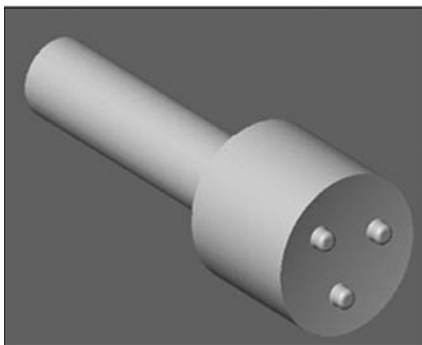
**Figure 3.** Tool materials clockwise from lower left: MP159 (a cobalt-nickel-chrome-moly alloy), H13 tool steel, and FerroTiC (35% TiC in an impact-resistant, iron-based tool steel)

virtually no wear. This project also investigated stirring ceramic particulate into steel substrates. In these cases, tools were composed of tungsten 25% rhenium.

Particle distribution within a surface- modified layer is dependent (among other things) on tool design. During FY 2004 this project investigated numerous tool designs in an effort to improve particle distribution and placement with a processed layer. Figure 4 shows tools of differing designs, including wide pins, narrow pins, scrolled shoulders, and multi-pin tools. The basic design philosophy is to create a wide stir zone (Figure 5) with a high degree of vertical mixing to drive the particulate from the surface to the deepest part of the processed region



**Step Spiral Tool**

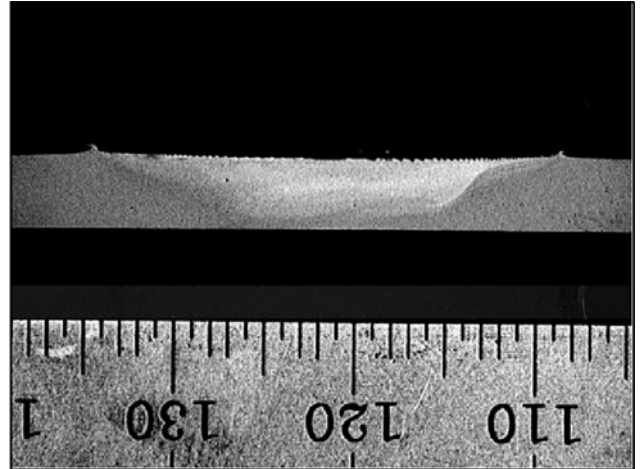


**Three Pin Tool**



**Three Paddle Tool**

**Figure 4.** Different tool designs produce different particle distributions and flow characteristics.



**Figure 5.** Wide stir zones can be created using three-pin tools. Wide zones are important to minimize passes required to process larger areas.

(Figure 6). Experimental work has shown that both the three-pin designs and the wide stepped spirals can achieve this goal.



**Figure 6.** Tool design affects material flow during FSP. Features on the tool surface can promote flow from surface to base of processed region and channel surface powders, for instance, to the pin root zone.



## Friction Stir Surface Processing

Much of the effort in FY 2004 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  $\text{TiB}_2$  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 (creep-resistant surfaces for engine applications).

from X-ray diffraction analysis. Despite the highly favorable Gibbs free energies for these reactions, only limited reaction products were formed. The large driving force for reaction with little reaction is a clear indication that the activation energy for the reaction was not satisfied. Higher temperatures decrease the activation energy, making spontaneous reaction more likely. In FY 2005, the project will investigate the FSRP process in higher-temperature processing environments like those found when processing iron- or titanium-based materials. In addition to higher temperatures, catalysts may also be employed as a means of lowering the activation energy. Additional work is planned on this as well as on other reaction systems, as outlined in Figure 9, including systems where the tool pin is a reactant and thermite-type systems.

Work was also initiated on a study of the potential for using FSP to process previously deposited thermal barrier coatings (TBCs). In many applications, TBCs can show reliability problems as a result of cracking and spalling of the coating at the

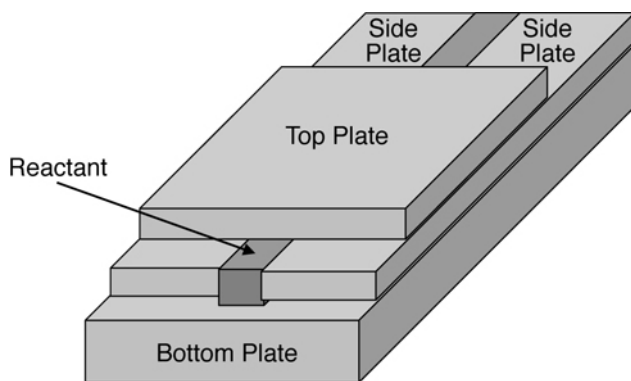
**Table 1.** Reaction modes

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

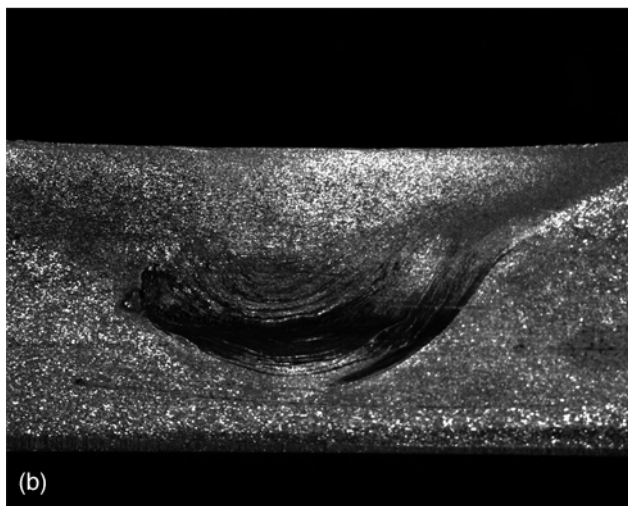
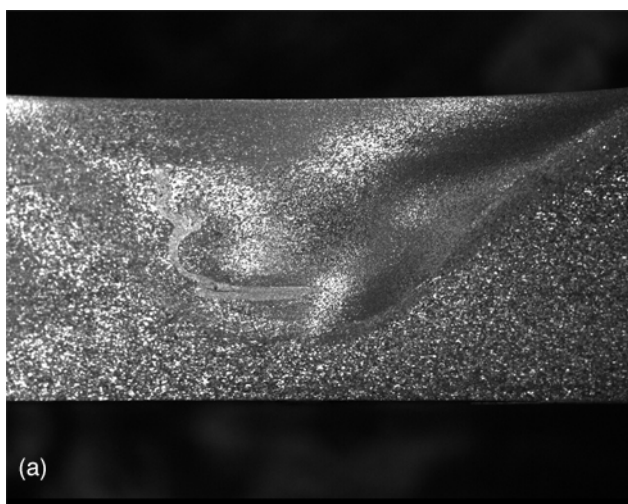
Work in FY 2004 concentrated on reactions in the aluminum system, as outlined in Table 1.

Reactant powders were placed between plates of 1100 alloy aluminum as shown in Figure 7. The assembly was then friction-stir-processed to produce a microstructure, as shown in Figure 8. The nugget region contains very finely dispersed precursor reactants but only a small amount of reactant product coating/substrate interface. FSP may be able to cre-

ate more robust coatings by disrupting the sharp interface and creating a graded structure. Issues with coefficient of thermal expansion mismatch may be reduced by creating this graded stir zone. Most TBC systems are in materials that are very difficult to FSP because of their hot hardness or lack of plastic properties needed to form good FSP microstructure.



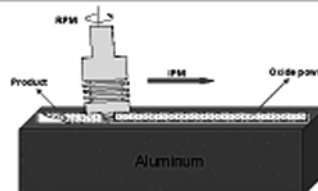
**Figure 7.** Reactant oxide powders are placed between plates, and the stir tool processes the entire thickness



**Figure 8.** Cross sections showing fully consolidated weld zone. FSRP zone is 3/8-in (9.52 mm) thick: (a) SiO<sub>2</sub> and (b) TiO<sub>2</sub>.

### Mode I

Base metal reacts with the oxide powder introduced during the friction stir welding process.



Reactions of interest:



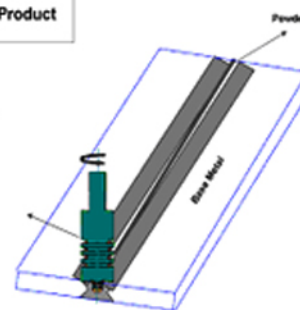
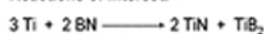
Note: non-oxide powders may also be used

### Mode II

The pin tool material reacts with the powder which is placed between two plates during the friction stir welding process.

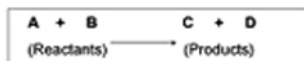


Reactions of interest:



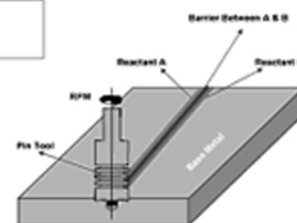
### Mode III

Two different materials react to form products. The two materials can be in powder or wire form.



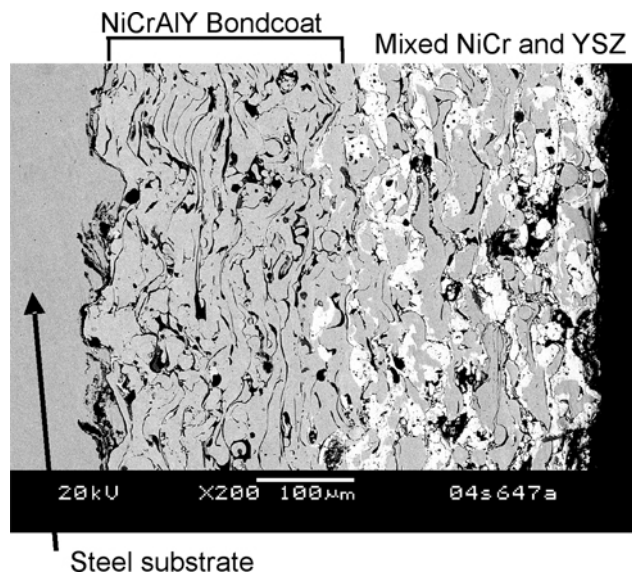
Reactions of interest:

All the reactions that were used for Mode I & II will be considered



**Figure 9.** Different modes of reactions and experimental work planned in the current program.

Most of the work in FY 2004 was directed at finding process conditions in which the coating, usually a zirconia-based material, could be successfully mixed into the substrate, usually a steel. Figure 10 shows typical flame spray coatings investigated in this study. These are usually characterized by a top coat of ceramic material underlain by a bond coat of NiCrAlY on a steel substrate. The project investigated various combinations of coating thicknesses

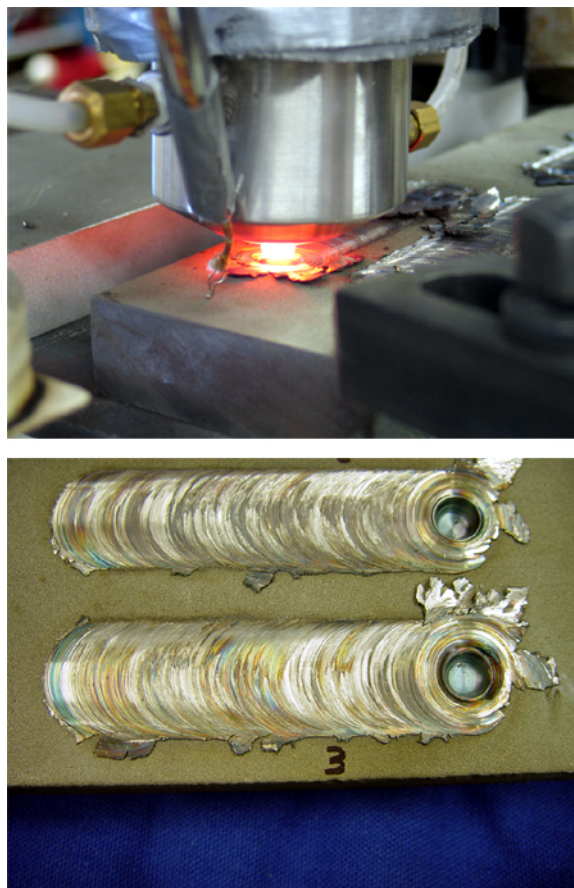


**Figure 10.** Typical flame spray deposited thermal barrier coating.

and graded metal ceramic coatings for their potential to be plasticized and processed using FSP. Figure 11 shows an example of a processed plate both during and after FSP. In some cases, especially with thick ceramic top coats, a large percentage of the top coat was chipped off during processing; but in other cases of graded or mixed ceramic/bond metal coatings, much of the coating was stirred into the nugget region.

Currently this project is investigating the thermal diffusivity/conductivity of these modified surfaces relative to the original coating; it is also investigating ways to better optimize the process so that less of the coating is lost during FSP.

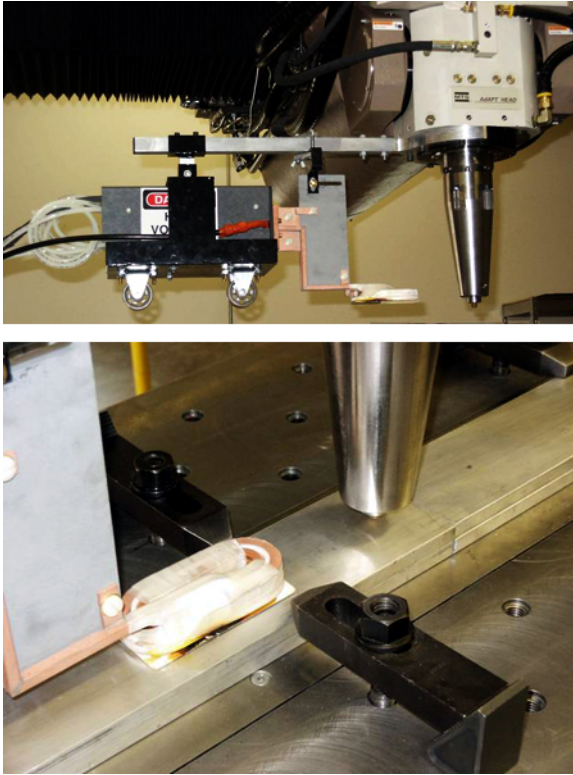
Research at the South Dakota School of Mines and Technology has shown that induction preheating of the substrate prior to FSP can have an important effect on process forces and perhaps on the quality of the weld in difficult-to-weld materials. Figure 12 shows the induction heater mounted on the spindle head of the friction stir machine. Figure 13 shows that for 1018 steel, induction preheating can reduce process forces throughout the weld or processed region length.<sup>1</sup> Reduced process forces can be correlated with lower tool wear and better material flow in the weld. Additional work is planned in FY 2005 to define the effects of induction preheating better in steel and difficult-to-process materials.



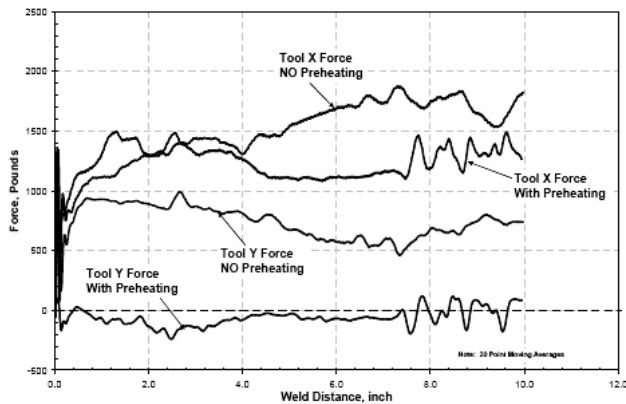
**Figure 11.** Zirconia-based thermal barrier coatings undergoing friction stir processing to mix coating into base material.

A final area investigated in FY 2004 was the applicability of FSJ/FSP to ferritic ODS alloys. These alloys have very good elevated-temperature creep properties resulting from the presence of fine dispersions of yttria in the steel matrix. One problem in using these materials for high-temperature applications is that they are very difficult to join. Few processes exist that can join these materials to other structures, or to themselves, without destroying the yttria dispersion and losing the high-temperature properties that make these materials attractive. Fusion welding can cause particle segregation, growth, or dissolution. FSP, however, is a solid state process and has the potential to join and process these alloys without destroying the yttria dispersion. Preliminary work during FY 2004 has shown that FSP can plasticize these alloys. Extruded tubes (25 mm in diameter with 3-mm wall thickness) of MA-957 with Inconel 625 inner rods were friction-stir-processed using a less than optimum



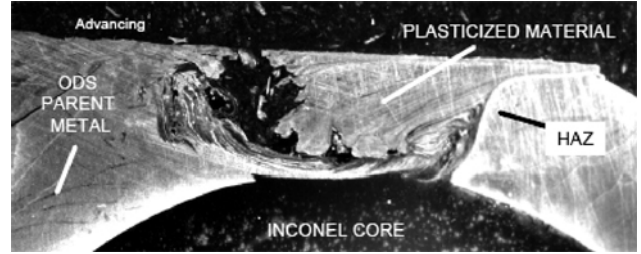


**Figure 12.** Induction pre-heating head mounted to FSJ machine spindle.



**Figure 13.** 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, Abergast, Allen 2004).

tool. Figure 14 shows plasticized material in the processed region. Transmission electron microscopy analysis and mechanical testing are under way to



**Figure 14.** Cross section of ODS alloy tube plasticized by friction stir processing. Processed region is not consolidated due to tool-part mismatch, but plasticized region shows nature of flow and confirms that ODS alloys can be processed using FSP.

determine the effect of FSP on the yttria dispersion and whether creep properties are preserved.

## 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, such as Al-MMCs and certain high-strength steels (including ODS), 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 new applications, and suggest new materials and engineered surfaces that can help deliver lighter and more fuel-efficient vehicles.

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