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FRICTION STIR WELDING AND PROCESSING VI

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Dedication

In recognition of his significant contributions to friction stir welding research, we dedicate this volume to Bill's memory.

Dr. "Bill" Arbegast was an enthusiastic champion of friction stir technology, a generous colleague, and supportive student mentor.

He will be greatly missed by many.

"Who is John Galt?"
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Editors

Rajiv Mishra is a Curators’ Professor of Metallurgical Engineering in the Department of Materials Science and Engineering at the Missouri S&T. He is also the Missouri S&T Site Director of the NSF I/UCRC for Friction Stir Processing and a Fellow of ASM International. His highest degree is Ph.D. in Metallurgy from the University of Sheffield, UK (1988). He has received a number of awards which include: the Firth Pre-doctoral Fellowship from the University of Sheffield, the Brunton Medal for the best Ph.D. dissertation in the School of Materials from the University of Sheffield in 1988, the Young Metallurgist Award from the Indian Institute of Metals in 1993, Associate of the Indian Academy of Sciences in 1993, the Faculty Excellence Awards from the University of Missouri-Rolla in 2001, 2002, 2003, 2004, 2005, 2006 & 2007. He has authored or co-authored 210 papers in peer-reviewed journals and proceedings and is principal inventor of four U.S. patents. His current publication based h-index is 33 and his papers have been cited more than 4000 times. He has co-edited a book on friction stir welding and processing, and edited or co-edited twelve TMS conference proceedings. He is the current chair of the joint commission for Metallurgical and Materials Transactions and serves on the Board of Review. He serves on the editorial board of Science and Technology of Welding and Joining, and Advances in Materials Science and Engineering.

Murray W. Mahoney has a B.S. (UC Berkeley) and M.S. (UCLA) both in physical metallurgy. Mr. Mahoney, consultant, has more than 44 years experience in physical metallurgy and related disciplines. Most recently, his work has centered on developing friction stir welding and friction stir processing. This work has led to the introduction of friction stir welding to join metals considered unweldable and advance FSW to higher temperature alloys. In addition, developments in friction stir processing have advanced superplasticity to very thick section structural Al alloys, enhanced room temperature formability, and improved mechanical and corrosion properties in cast Al and Cu alloys. These studies have resulted in a more complete metallurgical understanding of joining fundamentals, micro-

xiii
structural evolution, formability, and corrosion sensitivity associated with friction stir welding and processing. Mr. Mahoney has authored or co-authored over 90 publications and been awarded over 20 U.S. patents.

Yutaka Sato is currently an Associate Professor in the Department of Materials Processing at Tohoku University, Japan. He earned a Ph.D. in Materials Processing at Tohoku University (2001). His Ph.D. thesis was entitled "Microstructural Study on Friction Stir Welds of Aluminum Alloys." He participated in friction stir research of steels at Brigham Young University for a year in 2003. He is a member of Sub-commission III-B WG-B4 at IIW, which is a working group to build international standardization of friction stir spot based processes. His work has focused on metallurgical studies of friction stir welding and processing for more than a decade. He has obtained fundamental knowledge on development of grain structure, texture evolution, joining mechanism, behavior of oxide-layer on surface, properties-microstructure relationship, and so on. Recently, he has centered on developing friction stir welding of steels and titanium alloys, and new tool materials. He has received a number of awards including District Contribution of Welding Technology Award from Japan Welding Society in 2005, Kihara Award from Association for Weld Joining Technology Promotion in 2008, Prof. Koichi Masubuchi Award from AWS in 2009, Murakami Young Researcher Award from the Japan Institute of Metals in 2010, and Aoba Foundation Award in 2010. He has authors or co-authors more than 120 papers in peer-reviewed journals and proceedings.

Yuri Hovanski is currently a Research Engineer at Pacific Northwest National Laboratory. He earned a B.S. Degree in Mechanical Engineering at Brigham Young University, and then completed his M.S. degree in Mechanical Engineering at Washington State University. He is a member of Tau Beta Pi Engineering Honors Society, and actively participates in AWS and TMS serving on the forming and shaping as well as joining committees. He has participated in friction stir related research for more than a decade investigating weld formability, abnormal grain growth, and the influence of post-
weld microstructure and texture on mechanical properties. More recently, he has focused on the development of low-cost solutions for friction stir welding, introducing cost efficient solutions for thermal telemetry, new tool materials and production techniques for friction stir spot welding tooling, and utilizing thermo-hydrogen processing to aid friction stir welding of titanium alloys. He continues this effort today furthering the capability of friction stir spot welding in a variety of advanced high strength steel alloys, and recently introducing scribed tooling that enables lap welding of highly dissimilar materials. He actively reviews friction stir related literature for several publications and has documented his work in more than 25 publications.

Ravi Verma is a Staff Researcher in the Manufacturing Systems Research Laboratory at the General Motors Global Research and Development Center in Warren, MI. He has over 20 years of industrial and academic R&D experience in structural metallic alloys, processing, structure, properties, forming and simulation, with over 40 publications in archival journals and conference proceedings. More recently, he has focused on joining and durability of lightweight metallic structures, with particular emphasis on friction stir welding. He has interest in the influence of post-weld microstructure and texture on mechanical properties. Before joining GM, he worked as a research scientist at the University of Michigan, Ann Arbor, working in the area of deformation processing of high temperature metallic alloys and fiber-reinforced MMCs. He is a recipient of GM’s Charles L. McCuen, Chairman’s Honors, and “Boss” Kettering Awards. He holds a Ph.D. in Materials Science from the University of Bombay, India.
FRICITION STIR WELDING AND PROCESSING VI

High Temperature Materials I
DEVELOPMENT OF A COBALT-BASED ALLOY FSW TOOL FOR HIGH-SOFTENING-TEMPERATURE MATERIALS

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Keywords: Friction stir welding, Tool, Co-based alloy, Steels, Ti alloys

Abstract

The authors have developed a new friction stir welding (FSW) tool that enables welding of high-softening-temperature materials, such as steels, and titanium alloys. The new tool is made of a Co-based alloy strengthened by precipitating intermetallics, Co₃(Al,W), with an L1₂ structure at high temperatures. The Co-based alloy tool can be manufactured at a low cost through a simple production method consisting of casting, heat-treatment, and then machining. It exhibits yield strengths higher than 500 MPa at 1000°C, so that it might have great potential as a FSW tool for high-softening-temperature materials. In this study, the feasibility to use the Co-based alloy tool to friction stir weld various high-softening-temperature materials was examined. Changes in tool shape during FSW and the weld appearances produced with the Co-based alloy tool will be briefly shown.

Introduction

The tool material is critical in friction stir welding (FSW) of high-softening-temperature materials (HSTMs). The tool should meet significant requirements, i.e., it must maintain sufficient strength to constrain the material to be welded at the softening temperature, and also be resistant to fatigue, fracture, mechanical wear, and chemical reactions with both the atmosphere and the weld material [1]. To date, two classes of materials have been found that meet these requirements: refractory metal tools and superabrasive tools [1].

W-Re is well known as a representative material for fabrication of refractory metal tools. Alloys based on W-Re enable welding of Ti/Ti alloys and relatively thick steel plates. W-Re tools exhibit good fracture toughness, but often show a tendency to wear rapidly. A superabrasive tool made of polycrystalline cubic boron nitride (PCBN) is also used in FSW of steels. PCBN tools show better wear resistance than the refractory metal tools, but often experience unexpected cracking during FSW due to the low fracture toughness. To make up for disadvantages of two classes of tool materials, the composite tool material consisting of cBN and W-Re powders has also been recently developed [2]. This tool material has relatively good wear resistance and fracture toughness. However, this new tool material, as well as the W-Re alloys, is expensive. Thus, usage might be limited in practical production applications.

The authors have developed a new FSW tool material that enables welding of the HSTMs. The new material is a Co-based alloy with the γ/γ' microstructure. This alloy is strengthened by precipitating intermetallics, Co₃(Al,W), with an L1₂ structure. The yield strength is higher than 500 MPa at 1000°C. Since the new Co-based alloy can be easily cast and has good machinability,
this tool can be manufactured at a low cost through a simple production method consisting of casting, heat-treatment, and then machining. These attributes suggest that the new Co-based alloy tool might have significant potential as a low-cost FSW tool for HSTMs with relatively good performance. In preliminary trials, this tool exhibited acceptable performance in some HSTMs. In this manuscript, initial results are presented using the new Co-based alloy tool, i.e., changes in tool shape during FSW of the HSTMs and the weld appearance are briefly introduced.

### Co-based high-temperature alloy

Co and Ni have similar metallurgical characteristics. In general, however, it was known that Co-based alloys showed lower high-temperature strength than Ni-based superalloys, because the ordered intermetallic phases with the L1₂ structure, which is effective for high-temperature strengthening, could be stably only in Ni.

However, a ternary phase Co₃(Al,W) with the L1₂ structure, designated as γ', has been found in the Co-Al-W ternary system by the co-authors in 2006 [3]. The coherent γ/γ' microstructure of the Co-Al-W alloy is morphologically identical to that of a Ni-based superalloy [3,4]. The new Co-based alloy exhibits higher high-temperature strengths than the Ni-based superalloy, i.e., higher than 500 MPa at 1000°C. This is probably due to the higher strength of the Co₃(Al,W) γ' phase at temperatures higher than 800°C than that of the Ni₃Al γ' phase [5]. Additionally, the Co-based alloys are generally used in wear-resistant components at both room and elevated temperatures. The high strength and good wear resistance suggests that the new Co-based alloy with the γ/γ' microstructure might be suitable to use for FSW tools for HSTMs. Moreover, the new Co-based alloy shows good castability and machinability. The tool can be manufactured through simple methods consisting of casting using the lost-wax process, heat-treatment, and machining. These common manufacturing methods would be suited to the mass production of FSW tools.

![Figure 1. Appearance of the as-cast Co-based alloy (a) and a tool machined by standard methods (b).](image)

### Experimental procedures

A FSW tool was made using the new Co-based alloy strengthened by γ' Co₃(Al,W) with the L1₂ structure. 28 rods of the new Co-based alloy were produced by a lost-wax casting process
(Figure 1a) and then heat-treated to obtain the $\gamma'$ Co$_3$(Al,W) precipitates in the $\gamma$ matrix. The tool shape was machined on the new Co-based alloy by a standard machining method (Figure 1b). Appearance of the tool tip is shown in Figure 2. The tool design included a 15 mm diameter shoulder and an unthreaded pin with a length of 1.7 mm. The pin was tapered from 6 mm at the shoulder to 3.5 mm at the pin tip. FSW was conducted with 4 kinds of HSTMs: mild steel, ultrahigh carbon steel (Fe-1.4wt%Cr-1.0wt%C), commercial-purity Ti (cp-Ti) (grade 2), and Ti-6Al-4V. The welding parameters used with each HSTM are listed in Table 1. To minimize surface oxidation, argon shielding was employed around the tool during FSW. A 3° tilt was applied to the tool.

The tool appearance was examined before and after each FSW trial. Cross sections of the tool subjected to FSW were also investigated after FSW trials on mild steel and Ti-6Al-4V. Microstructures of the tool on the cross section were observed by optical microscopy without etching.

Table 1. Welding parameters used with each material

<table>
<thead>
<tr>
<th>Materials to be welded</th>
<th>Rotational speed (rpm)</th>
<th>Travel speed (mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mild steel</td>
<td>250</td>
<td>500</td>
</tr>
<tr>
<td>Ultrahigh carbon steel</td>
<td>200</td>
<td>60</td>
</tr>
<tr>
<td>cp-Ti</td>
<td>200</td>
<td>100</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>200</td>
<td>100</td>
</tr>
</tbody>
</table>

Results and discussion

Mild steel

The weld appearance of mild steel is presented in Figure 3a. The new Co-based alloy tool produced a smooth and shiny surface on the mild steel. The weld included a small defect at the bottom of the stir zone, which could be eliminated by optimization of the tool shape. Appearances of the tool tip subjected to several weld lengths are shown in Figure 3b. The tool shape hardly changed after FSW a 0.4 m length. No large variation of the tool shape was seen for
weld lengths less than 10 m. For weld lengths >10 m, both shoulder wear and pin deformation were detected, as shown in the tool appearance subjected to 45.7 m FSW, Figure 3b.

A cross section of the tool subjected to 45.7 m FSW is shown in Figure 4a. Peripheries of the pin and shoulder were obviously worn during FSW. Optical micrographs of the shoulder periphery and the pin tip are shown in Figures 4b and c, respectively. The as-cast microstructure was deformed near the surface where the mild steel contacted during FSW. This implies that the tool wear occurred mechanically. Moreover, cracking is found at the root of the pin. Cracking at this location has been observed previously in other tool materials [6]. In this Co alloy, the cracks propagated along the solidification cell boundaries. Based on this observation, the cracking could possibly be prevented by optimization of the lost-wax process, i.e., refinement of the solidification cell might be effective.

Figure 3. (a) Weld appearance of mild steel and (b) appearances of Co-based alloy tools subjected to several weld lengths.

Figure 4. (a) Cross section of a Co-based alloy tool subjected to 45.7 m of FSW in mild steel and (b)-(d) optical micrographs of selected regions on the cross section shown by b-d.
Ultrahigh carbon steel

The weld and tool appearances of ultrahigh carbon steel are shown in Figure 5. A defect-free weld with a smooth surface was obtained in this steel. A FSW weld length of 150 mm leads to no large change in tool shape, but the ultrahigh carbon steel caused more wear than the mild steel. Wear should be related to a difference in mechanical properties between mild and ultrahigh carbon steels. Examination to clarify correlation between tool wear and mechanical properties of the welded material is in progress.

![Figure 5. Weld appearance of ultrahigh carbon steel, and macroscopic overviews of the tool before and after FSW.](image)

Cp-Ti

FSW led to a rough surface in cp-Ti compared to mild and ultrahigh carbon steels. A small defect was seen in the stir zone. The rough surface is attributable to the low thermal conductivity of Ti, which may be solved by optimization of either the tool shape or process [7]. Although the weld surface was rough in cp-Ti, the tool shape hardly changed. Some tool materials that contain elements having a high affinity with Ti, e.g., PCBN, are unusable to friction stir weld Ti alloys because chemical wear occurs during FSW [8]. This result implies that the new Co-based alloy tool has a sufficient resistance to mechanical and chemical wear during FSW of Ti and Ti alloys.

![Figure 6. Weld appearance of Ti-6Al-4V.](image)
The weld appearance of Ti-6Al-4V is shown in Figure 6. A defect-free weld with a very smooth and shiny surface was produced in Ti-6Al-4V. The tool appearances before and after 200 mm FSW are presented in Figure 7a. No macroscopic change in tool shape is seen. A cross section of the tool subjected to 200 mm FSW is shown in Figure 7b. The pin is hardly deformed or worn, but the shoulder surface has become rough. Optical micrographs of regions c and d are shown in Figures 7c and d, respectively. Selective areas are preferentially worn, but the reason for the selective wear could not be clarified in this study. Examination of the wear mechanism based on the metallurgical observations is in progress.

Summary

In this study, the feasibility of a cost-efficient Co-based alloy tool with the γ/γ' microstructure for some HSTMs was briefly shown. The new Co-based alloy does not contain any expensive alloying elements, and a tool can be manufactured through simple methods suited to mass production. Even if tool wear or deformation occurs, the tool shape can be easily produced by re-machining. The preliminary FSW trials established that the tool successfully produced welds in some HSTMs without large variation of tool shape. Currently, the FSW trial of thick steel plates and optimization of the chemical composition of the Co-based alloy are ongoing. Further results will be reported in the separate papers.
Acknowledgements

The authors are grateful to Dr. Y. Takaku and Mr. A. Honda for technical assistance. Financial support from the Japanese Ministry of Education, Science, Sports and Culture with a Grant-in-Aid from the Global COE Program in Materials Integration International Center of Education and Research at Tohoku University is also gratefully acknowledged.

References


FRICITION STIR WELDING OF ALLOY 22

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Keywords: Alloy 22, Friction stir welding, TCP phases

Abstract

The main objective of this work is to characterize and compare Alloy 22 weld microstructures formed in friction stir (FS), gas tungsten arc (GTA), and electron beam (EB) welding. Topologically closed packed (TCP) phases were identified in all the as-welded condition. TCP phases that precipitated in the FS weld nugget were extremely small (~50 to 300 nm) compared to those in the EB and GTA welds. Compared to the parent material the FS weld nugget microhardness increased from (~206 HV) to (~327 HV). Only a slight increase in microhardness was observed for the EB and GTA welds. The area fraction of the TCP phases in FS weld nugget increased with aging temperature and time. The activation energy calculated for the TCP phase growth in friction stir welds was determined to be ~252.5 kJ/mol.

Introduction

Alloy 22 (NO6022), also known as Inconel 622 and C 22 is a nickel-chromium-molybdenum-tungsten alloy. It has excellent corrosion resistance and was proposed as the outer barrier material for packaging of nuclear waste for disposal at Yucca Mountain, Nevada, USA [1]. The waste package design called for a 2-cm thick outer barrier made of Alloy 22 and a 5-cm thick 316 stainless steel (UNS S31600) inner vessel [1]. The inner barrier offers necessary structural support and mechanical strength; whereas the outer barrier provides the necessary corrosion resistance. The excellent corrosion resistance of Alloy 22 results from the presence of molybdenum and chromium [2]. This alloy has a single phase, face-centered cubic structure in the mill-annealed condition. Other phases may precipitate with time and degrade both mechanical properties and corrosion resistance of the alloy [3]. One of the concerns for the waste package integrity is the long-term corrosion resistance of the closure welds [4-11]. During the first several hundred to thousand years of service in nuclear waste disposal, the temperature of the waste container is expected to reach a maximum temperature between 250 °C and 300 °C. Therefore, the impact long exposure times on FSW microstructure at these temperatures must be considered.

Previous work on Alloy 22 suggests the formation of intermetallic tetrahedral (or topologically) closed-packed phases (TCP) during solidification [12]. These molybdenum-rich, phases are formed during solidification. The orthorhombic "P" phase is stable at 700 °C to 800 °C [8]. The rhombohedral "μ" phase is stable between 600 °C and 700 °C [8]. The tetragonal "σ" phase is stable between 800 °C and 1000 °C [8]. The precipitation of these TCP phases could decrease the corrosion resistance of Alloy 22 and could have an impact on the mechanical properties. GTA
welding is employed for the fabrication of the containers. GTA welding involves melting of the base materials and produces a cast and segregated microstructure in the weld nugget. The TCP phases are present in the as-welded GTA weld nugget [6, 13]. Solution annealing heat treatment eliminates these phases [6]. However, this heat treatment is not possible for closure welds because the contained nuclear waste cannot be heated to more than 350 °C [3, 13-14]. This segregated microstructure in the weld nugget could impact both the mechanical and corrosion properties. Since friction stir welding is a solid state joining process, the problems associated with solidification and segregation could be avoided. In this investigation the microstructures in Alloy 22 FS welds were compared with EB and GTA welds. Alloy 22 FS welds were heat treated to determine the effect of aging temperature and time on the growth rate of the TCP phases.

**Experimental**

The friction stir welds were made using an MTS-Istir-10® FSW system equipped with a Megastir® water-cooled, high-temperature pin tool (HTPT) adapter. High-temperature pin tools made of polycrystalline boron nitride (PCBN) were used to make bead-on-plate FSW welds in 6.35-mm thick Alloy 22 plates. PCBN pin tools with a convex scrolled shoulder, stepped spiral (CS4) pin design were used. A radio-frequency telemetry system on the tool adapter allowed monitoring of the weld temperature immediately above the pin tool shoulder. The tools and the Alloy 22 plates were shielded with argon gas during welding to prevent oxidation. The experimental setup and a photograph of the PCBN pin tool are shown in Figure 1.

![Figure 1: Friction stir weld setup and PCBN-CS4 pin tool](image)

After welding, the plates were sectioned perpendicular to the welding direction and prepared for metallographic examination using standard metallographic polishing procedures. The polished specimens were electrolytically etched with a solution consisting of 5 g oxalic acid and 95 ml of 37% hydrochloric acid at 5 V for a few seconds. The specimens were aged at 650°C, 705°C and 760°C for 5, 21 and 60 hours in a tube furnace under an argon atmosphere. Growth of the TCP phases were determined from the area fraction measurements. Micrographs for each specimen were acquired at 20,000X using a scanning electron microscope (SEM) in backscattered electron detector mode. The difference in the chemical composition between the TCP phases and the matrix facilitated identifying the TCP phases [14]. The amount of electrons backscattered in a given volume of material is directly proportional to the atomic number (Z) of the material [14]. Therefore, the molybdenum rich TCP phases show up with a higher intensity (bright) against a dark matrix.