Research Experience for Undergraduates

National Science Foundation

# Comparison of Friction Stir Welded MA956 Superalloy with and without Post Weld Heat Treatment

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## Abstract

Friction stir welding (FSW) is conducted on the oxide dispersion strengthened (ODS) ferritic stainless steel superalloy MA 956. Post weld heat treatments (PWHT) at varying temperatures and times are performed on the FSW and parent material regions of MA 956 in order to explore the properties of the microstructure in the parent material and the FSW after the annealing process. Vicker's hardness testing, grain size analysis using the line-intercept method, and SEM analysis are conducted to examine the microstructure and investigate the effect of annealing on this superalloy.

## Background

The research conducted on FSW MA956 ODS superalloy is used to portray the growing need and importance of using solid-state welding techniques as opposed to arc welding techniques and how the friction stir weld responds to the PWHT. Friction Stir Welding (FSW) is a solid-state welding process. This means no melting is involved, so the microstructure can retain most of its parent-material properties and thus produces a much stronger and safer joint for applications involving high temperature and radiation. In arc welding, the joint strength of a weld is greatly reduced in comparison to the parent material and it uses costly filler metal, expensive consumables, and shielding gas. No fumes are emitted in FSW since no melting is involved, so the overall FSW process provides a safer environment for trained workers whose jobs are to maintain and operate the FSW machine without costly safety equipment.

For our experiment, the best weld produced on the MA956 superalloy was performed at 600 RPM, 0.5 IPM under an argon atmosphere in order to decrease the likelihood of oxidization of the material.

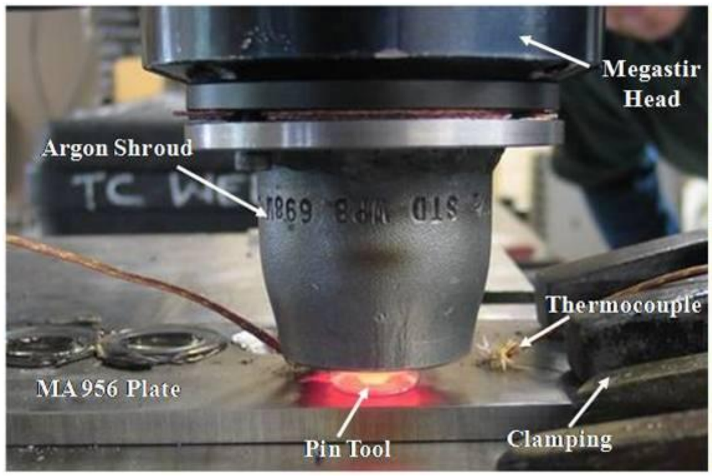


**Figure 1: The MTS ISTIR 10 Gantry Intelligent Stir Welding machine's W-Re pin tool is pictured welding our MA956 parent material.**

## Introduction

The oxide dispersion strengthened (ODS) INCOLOY® MA956 superalloy is heat, corrosion, oxidation, carburization, radiation, and creep-resistant up to temperatures of 1300°C during prolonged exposure [1,4,5]. MA956 is composed of (Fe) (20%Cr) (4.5%Al) (0.5%Ti) (0.5%Y2O3) (in wt %) and is made by the International Nickel Company (INCO) [2]. It was provided for our research by the Los Alamos National Laboratory (LANL). This ODS superalloy is produced by dispersing fine $Y_2O_3$ (yttrium oxide) particles into the ferritic matrix by a mechanical alloying (MA) technique, such as ball milling. The purpose of the distribution of the yttrium oxide is to increase the creep strength, thus providing greater strength at high temperatures, and to enhance the mechanical strength and high stability of the microstructure [2,7]. MA956 is preferable for use in nuclear reactors, advanced energy conversion systems, high-temperature shields, and many other high-temperature applications [3]. MA956 has a low carbon concentration in order to avoid the formation of titanium carbides, which are very brittle [1]. The ODS alloy also has a large amount of aluminum and a high concentration of chromium in order to be more resistant to oxidation [1]. Due to its columnar grain structure, MA956 is more creep-resistant than other alloys that have an equiaxed grain structure [3,4].

In this study, MA956 is friction stir welded (FSW) at 600 RPM and travels at a traverse speed of 0.5 inches per minute (IPM). After microstructural characterization of the initial FSW samples, a PWHT is performed in order to study the effects of annealing on the properties and microstructure of the FSW region of MA956.

## Broader Impact

Friction Stir Welding (FSW) is a solid-state welding process - no melting occurs. According to the results, the ODS alloy microstructure can retain 80-90% of the parent-material hardness. FSW produces a much stronger and safer weld joint than conventional arc welding for applications involving high temperature and radiation and no hazardous fumes are emitted. With no fumes, the FSW process is safer and does not require costly safety equipment, whereas conventional arc welding uses costly filler metal, consumables, and shielding gas.

FSW is in the technological future for ODS alloys due to the fact that there is no melting. When melting occurs, the oxide particles can be emitted from the alloy's microstructure. This defeats the purpose of mechanically alloying the stable oxide particles into the material's chemical composition in the first place – so it is important to retain these ODS particles. These stable oxide particles are what make the material capable of resisting creep at high temperatures, thus making for a safer, stronger material. There are few solid-state welding alternatives that offer the strength and cost-efficiency that FSW offers while retaining the important chemical compositions making up many new and improved materials.

## Procedures

The MA956 is welded using the MTS ISTIR 10 Gantry Intelligent Stir Welding machine – or FSW machine - at 600 RPM, 0.5 IPM under an argon atmosphere.

Samples are cut from the 600 RPM weld region and the parent material region using a DoALL band saw.

**Figure 2: The DoAll band saw is pictured with the wax that lengthens the life of the band saw's blade, our MA956 parent material, and a pusher safety tool.**

Once the samples are cut, light grinding of the samples takes off excess debris left over from the sawing process. Once light cleaning is performed using soap and water followed by a supersonic bath, the FSW and parent material samples are mounted using a cold mount. The grinding process is performed using 240 grit and goes up to 1200 grit. For polishing, a diamond solution of 3 micron diamond is used on Nylon; followed by a 1 micron diamond solution used on Lecloth; and then 1 micron colloidal silica is used on an Imperial cloth as a finishing polish. Thorough cleaning is required between each step of the grinding and polishing process in order to prevent contamination.

Once all the samples are prepared for examination, an electrolytic etch consisting of 30% HCl and 70% Ethanol at 1 volt and 2.5 amps is used for approximately 8-12 seconds in order to successfully reveal the grain boundaries. It is important in this study to research the grains in order to see if and how heat treatments affect the grains in the FSW-nugget region differently than the parent material region of MA956. The nugget region is the swirled region created by the pin tool of the MTS ISTIR 10 Gantry Intelligent Stir Welding machine. A photo of the weld nugget region is shown in figure 4.

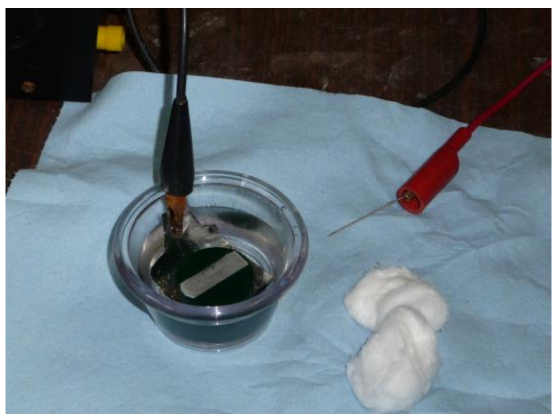

**Figure 3: Electrolytic etching is performed on the FSW region of our MA956 sample.**

The etched samples clearly showing the grain boundaries are then observed to study the microstructure of the nugget region in the FSW specimen. An optical microscope is used to take pictures of the microstructure at 20X magnification. Each of the four zones of the nugget region of a friction stir weld is examined by the micrographs taken. The four zones are: advancing side (zone 1), retreating side (zone 2), arm (zone 3), and swirl (zone 4). Micrographs are important in order to examine grain size and structure, while macrographs are taken in order to see a broader range of the FSW region. The macrographs show a broader view of the weld nugget and are good for examining hardness versus distance over the nugget region. After all necessary micrographs and macrographs are taken, hardness testing is performed.

The Vicker's Hardness Test is performed on both the parent material and the weld nugget region. It is important to note that on the FSW sample, the hardness values are taken in a straight line across the weld starting with the parent material closer to the advancing side (zone 1) and ending with the parent metal closer to the retreating side (zone 2). It is important to study the hardness within the nugget region as well as the region surrounding the nugget in order to visualize how the weld affects the properties of our MA956 material. These hardness values can then be compared to the values from the base material to see if the parent material surrounding the nugget is influenced by the weld.

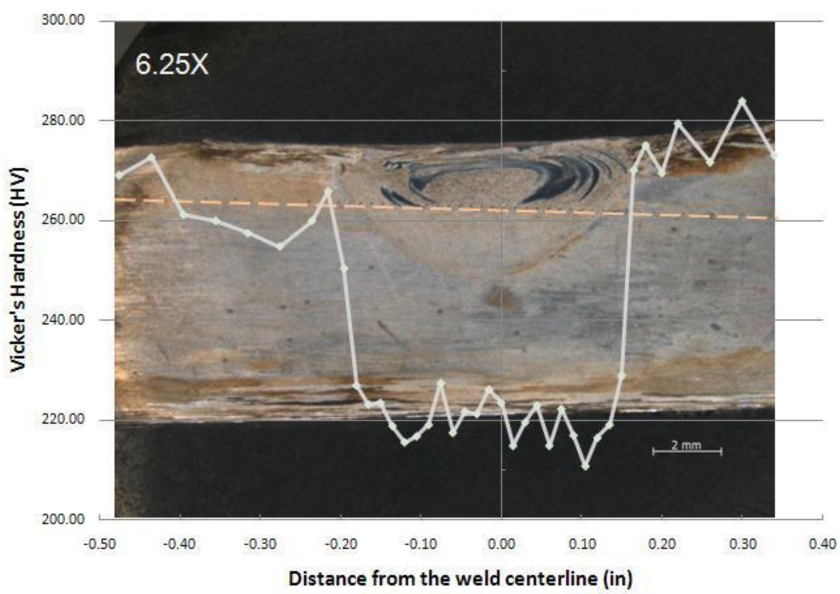


**Figure 4: MA956 weld nugget region with Vicker's hardness test values shown in comparison to the macrograph of the 600 RPM, 0.5 IPM at 6.25X magnification.**

New samples are cut using the DoALL band saw from the 600 RPM weld in order to have samples to study for the PWHT. The PWHT is performed on both the FSW and parent material regions of MA956.

The samples are put into a vacuum tube furnace in argon gas at 1000°C for 1 and 5 hours, and 1300°C for 1 and 5 hours. The furnace setup involves a cooling system for the ends of the glass tubes so the tubes are less likely to break, an alumina furnace boat to hold the samples within the furnace, argon gas to lessen the probability of oxidation, and thermocouples to moderate and check the temperature of the furnace.

Once the samples are prepared with the specified heat treatments, they are mounted, ground, polished, and etched just like the samples that did not have the PWHT explained before. They are also characterized similarly to the samples without the PWHT.

**Figure 5: ATS tube furnace running at 1300°C with water cooling system.**

## Results

- **Hardness Testing**
- **Grain Size Analysis**
- **SEM**

## Hardness Testing

Hardness testing in the samples without any heat treatments revealed a slight decrease in hardness in the FSW region. According to the graph, the FSW retained approximately 80-90% of the hardness compared to the surrounding parent material.

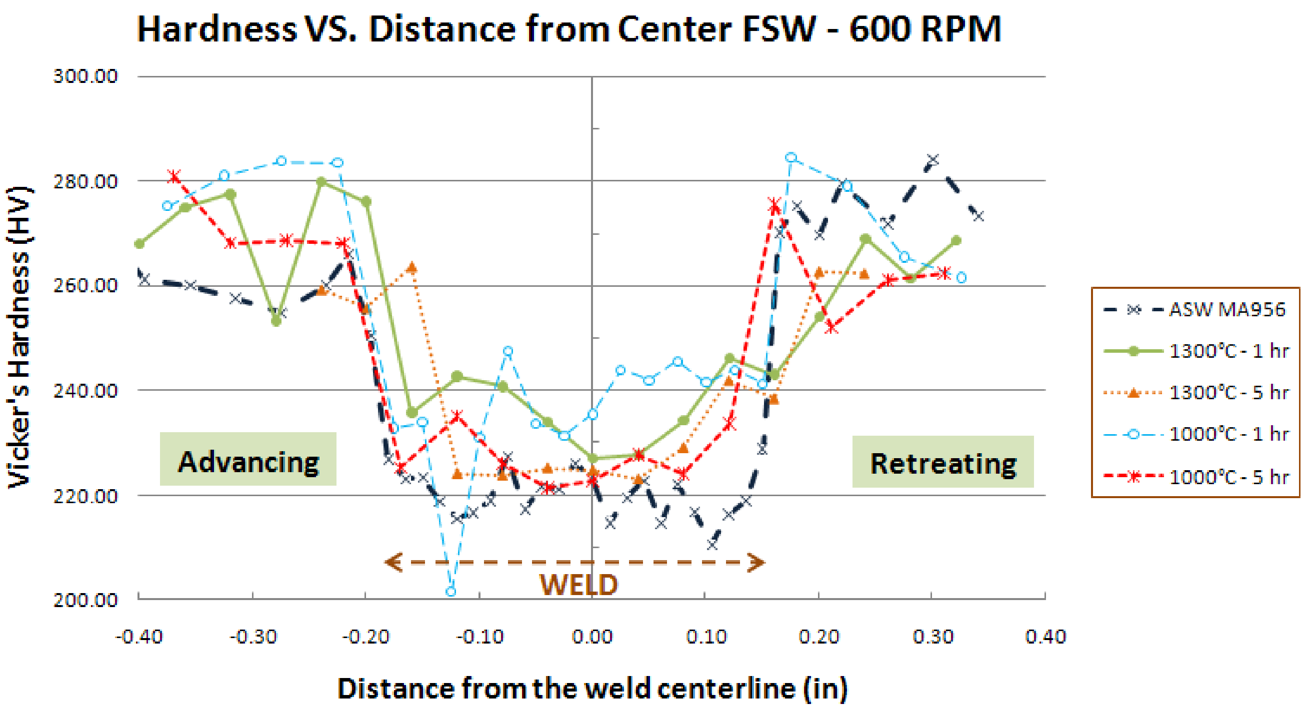


**Figure 6: Hardness results across a FSW nugget region for the PWHT samples at 1000°C and 1300°C for 1 and 5 hours compared to the as welded (ASW) sample that is not heat treated. MA956 is FSW at 600 RPM and 0.5 IPM.**

In figure 6, all the hardness values are very similar. This is important because it means that the FSW region and the surrounding region of MA956 can retain most of its strength at high temperatures for prolonged periods of time. This information can be used to help judge the safety of the MA956 material with a FSW for the high-temperature applications for which it is created.

### *Grain Size Analysis*

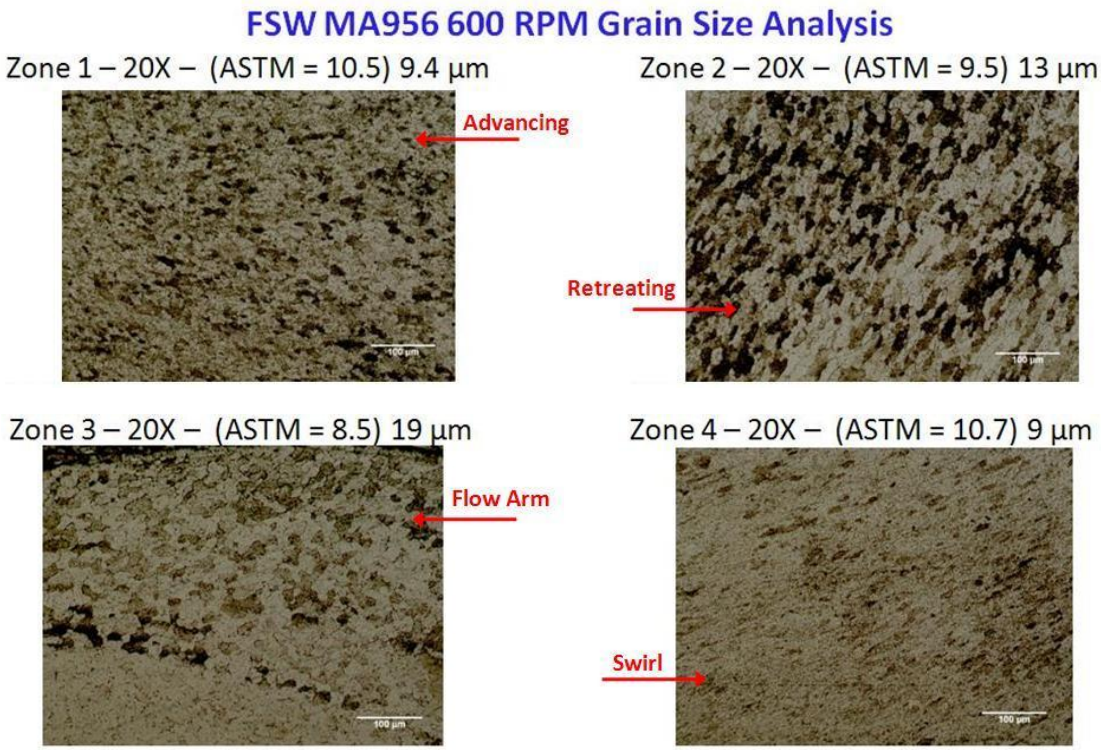


**Figure 7: MA 956 600 RPM FSW nugget region without PWHT. Four main zones are represented in the nugget region and are labeled accordingly, all magnifications at 20X.**

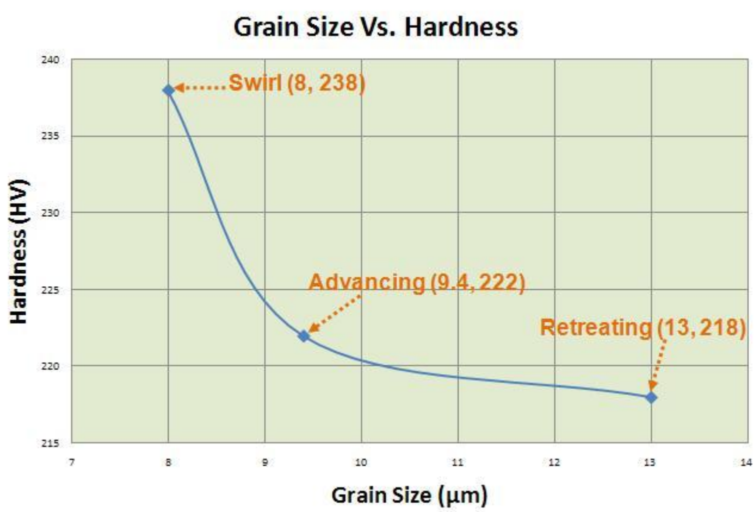


**Figure 8: MA 956 600 RPM FSW nugget region without PWHT – Average hardness results in three of the four zones of a FSW nugget region compared to the average grain size results using the ASTM line-intercept method. *Flow Arm zone was not included due to varied grain size and hardness measurements.**

As figure 8 portrays, when the grain size increases in the MA956 specimen with no PWHT, the hardness decreases. In the analysis of several micrographs not pictured in this report, it was observed that after the PWHT very little to no grain growth occurred in the weld nugget region of MA956, though it seemed that there was significant grain growth in the parent material surrounding the weld nugget. A necessary step to take for future work is to analyze the grain size and structure in the PWHT samples to see if the general trend for grain size and hardness still exists.

***SEM***

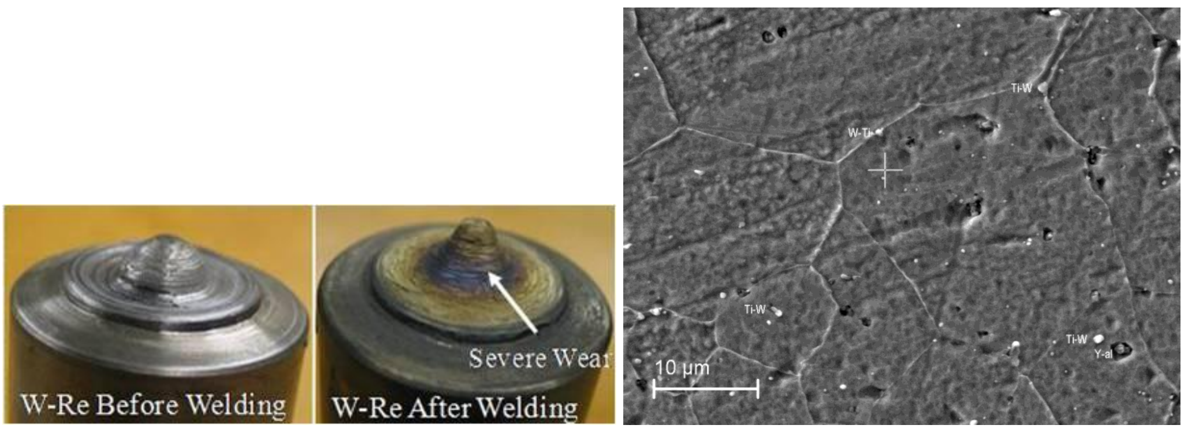


**Figure 9: Right: SEM at 5000X magnification of MA 956 600 RPM FSW nugget region PWHT at 1300°C showing W-Ti particles. Left: Visibly, severe wear occurred to FSW pin tool at 900 RPM. The micrograph on the right was taken in the flow arm region of the nugget zone and because there are W-Ti particles, this shows that not only was there pin tool wear in the 900 RPM weld, but in the 600 RPM weld too.**

## Discussion

One issue faced was whether to use an electrolytic or a chemical etch. An electrolytic etch that did not work consisted of 40% Nitric Acid and 60% distilled water. Two different chemical etchants were tried and neither produced sufficient results. One unsuccessful chemical etch used 95% HCl, 5% $H_2SO_4$, and 3% $HNO_3$. The other unsuccessful chemical etch used 75% HCl and 25% $HNO_3$.

According to the Vicker's hardness test results, the FSW region of MA956 will withstand post weld heat treatments up to 1300$^{o}$C for 5 hours and still retain a majority of its hardness. It appears that the grains located in the nugget region of the FSW do not change in size with applied PWHT, whereas the grains in the parent material surrounding the nugget grow significantly. It is interesting to point out that even though the grains grow significantly in the parent material region there is very little change in the hardness values.

In figure 6, the hardness values increase slightly from the as-welded condition (ASW) with increased annealing temperatures and varying times. Further research is necessary to determine whether any phase changes occurred or if precipitation of the material occurred within the FSW in order to produce the higher hardness test results.

## Conclusions

FSW is successful in making a weld in MA956 that is superior to conventional arc welding because the solid-state welding process of FSW retains the ODS particles, thus keeping the important creep-resistant structure that makes up MA956.

The hardness values in the FSW region of MA956 do not show significant change – only very slight change - when the PWHT has been performed. Looking at figure 6, it can be speculated that higher annealing temperatures from 1000$^{o}$C to 1300$^{o}$C annealed for one to five hours produce higher hardness values for MA956 within the FSW region aside from very few exceptions, shown on the graph.

The SEM micrographs showed that pin tool wear occurs at 600 RPM in the flow arm zone of the weld nugget due to the W-Ti particles found. Tungsten makes up the composition of the pin tool, but tungsten is not part of the chemical composition of MA956. Therefore, the tungsten within our MA956 ODS superalloy must have come from the pin tool. Tungsten was only found in the flow arm region near the top of the weld nugget where the pin tool primarily spins.

For future work, it is necessary that grain size analysis be performed for the PWHT samples of MA956 in the FSW region. It is important to compare the hardness values with the grain size structure to see if there is any correlation. Transmission Electron Microscope (TEM) analysis should be performed in the future in order to study the particles in the FSW and to explore particle stability after the PWHT.

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