



We are Nitinol.™

## **Friction Stir Processing of Nitinol**

Blair London, Jennifer Fino, Alan R. Pelton, and Murray Mahoney

Friction Stir Welding and Processing III, eds., K.V. Jata, M.W. Mahoney, R.S. Mishra, and  
T.J. Lienert, TMS, Warrendale, PA 67-74 (2005).

2005

---

[www.nitinol.com](http://www.nitinol.com)

47533 Westinghouse Drive Fremont, California 94539 t 510.683.2000 f 510.683.2001

## FRICITION STIR PROCESSING OF NITINOL

Blair London<sup>1</sup>, Jennifer Fino<sup>2</sup>, Alan Pelton<sup>2</sup>, Christian Fuller<sup>3</sup>, Murray Mahoney<sup>3</sup>

<sup>1</sup>Materials Engineering Department, California Polytechnic State University, San Luis Obispo, CA 93407, USA

<sup>2</sup>Nitinol Devices and Components, 47533 Westinghouse Dr., Fremont, CA 94539, USA

<sup>3</sup>Rockwell Scientific Company, 1049 Camino dos Rios, Thousand Oaks, CA 91360, USA

Keywords: Nitinol, Friction Stir Welding/Processing, Cubic Boron Nitride

### Abstract

Nitinol, a shape-memory alloy, is difficult to fusion weld to itself and to other metals. This can make certain post-weld processing operations expensive and can limit applications for the alloy. Void-free friction stir processed zones were produced in 6.35-mm (0.25-in) thick Nitinol plates using polycrystalline cubic boron nitride and tungsten-rhenium tool materials. Friction stir processing was done, since producing a processed zone is virtually equivalent to producing a welded joint. The processed zone microstructures were documented with optical and scanning electron microscopy. The austenitic and martensitic transformation temperatures of the processed regions were measured using differential scanning calorimetry. The nugget region showed a slightly reduced average grain diameter (33  $\mu\text{m}$ ) from the base metal (43  $\mu\text{m}$ ). The austenite finish transformation temperature was lower in the processed regions (-22.5°C) compared to the base metal (15.0°C). The 6.35-mm (0.25-in) thick friction stir processed plate was successfully hot-rolled at 850°C to a final thickness of 1.55 mm (0.061 in) for a 76% total reduction. The results of tensile tests on the processed plus rolled sheet indicated a slight increase in strength in the processed regions; likely because of grain size refinement, and no decrease in ductility compared to the rolled unprocessed sheet. The friction stir processed Nitinol retains its superelastic and shape memory properties, indicating that a friction stir welding process is feasible for Nitinol.

### Introduction

Nitinol is an alloy composed of near equiatomic proportions of nickel and titanium that exhibits extraordinary properties such as superelasticity and a shape memory effect [1]. Nitinol undergoes a diffusionless, martensitic phase transformation associated with a specific transformation temperature and a reversible change in crystal structure. The phase transformation occurs between a high temperature austenite phase and a low temperature martensite phase [2]. The "loading stress plateaus" for both austenite and martensite strongly depend on the transformation temperature, test temperature, and thermomechanical processing. In general, the martensite plateau is on the order of 100 MPa, whereas the austenite plateau can range from less than 200 MPa to greater than 1000 MPa. This extreme range for the austenite phase is quite predictable and follows the Clausius-Clapeyron stress rate function,  $d\sigma/dT$ , of approximately 6 MPa/K. Both martensite (shape memory) and austenite (superelasticity) can recover up to 10% strain. Although Nitinol is considered to be an intermetallic compound, it is capable of elongation to failure of over 50% and tensile strength of 1500 MPa [3]. In addition, the material is exceptionally biocompatible and corrosion resistant [4].

The austenitic and martensitic transformation temperatures can be determined by differential scanning calorimetry (DSC) as indicated in Figure 1. The lower curve, measured during sample heating, shows the austenite phase transformation beginning at the austenite start temperature ( $A_s$ ) and completed at the austenite finish temperature ( $A_f$ ). The upper curve shows how the martensite phase transformation takes place during sample cooling. The transformation temperature of Nitinol is commonly listed as the alloy's  $\Delta T$  temperature.

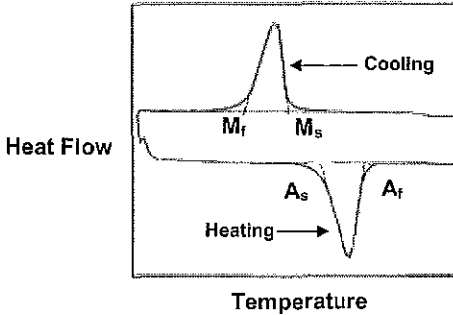


Figure 1. Typical differential scanning calorimetry (DSC) curve for Nitinol showing the austenite and martensite transformation temperatures.

The austenitic phase exhibits an ordered B2 crystal structure and the martensite phase has a highly twinned, close-packed monoclinic (B19') structure [5]. The numerous crystallographic pathways associated with Nitinol's phase transformations allow it to exhibit superelasticity and shape memory ability [1, 2, 6].

Unique properties of Nitinol have led to applications ranging from actuators to eyeglass frames [7]. One of the most common uses of Nitinol is in the production of biomedical devices such as arterial stents. A stent is a wire mesh tube inserted into an artery of a patient suffering from cardiovascular disease (Figure 2). The stent permanently props open the artery after it has undergone angioplasty, or the removal of plaque from the arterial walls [8]. Self-expanding Nitinol stents are machined from thin-walled tubes and are commonly used in peripheral vessels such as the carotid arteries [4].

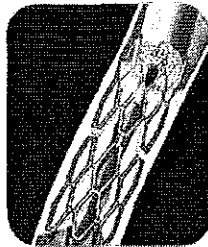


Figure 2. A wire mesh stent in place in an artery [9].

The current process of producing thin-walled Nitinol tubes involves gun drilling a 13 to 38 mm (0.512 to 1.50 in) diameter bar and then tube drawing to the needed outer tube diameter of 0.5 to 6.4 mm (0.020 to 0.252 in). Gun drilling is wasteful (25% loss in material) and therefore is a

major expense in the cost of stents and other thin-walled Nitinol medical devices. Welding Nitinol sheet into a relatively thick-walled tube that subsequently could be tube drawn to the proper wall thickness would eliminate the gun drilling step in stent manufacture. However, welding Nitinol continues to be a challenge using conventional joining techniques [10, 11].

Significant research has been done to determine welding process parameters to allow Nitinol to be successfully welded. Investigators have used fusion welding to join Nitinol; however, the cast microstructure has little ductility. Solid-state joining methods have been developed for Nitinol such as diffusion bonding. However, the pressure, temperature, and time parameters used during diffusion bonding must be highly controlled to ensure that Nitinol does not melt during the process [12].

The lack of melting during friction stir welding makes it a logical candidate for joining Nitinol. The present study investigated the feasibility of friction stir welding Nitinol by first producing friction stir processed regions in thick Nitinol plate. To simulate the tube drawing process that welded Nitinol material would have to undergo, hot rolling studies were conducted on friction stir processed Nitinol plate.

### Experimental Procedure

The starting material for this investigation was 6.35-mm (0.25-in) thick, fully annealed Nitinol plate (49.2 % Ti 50.8 % Ni, trace elements < 100 parts per million). The coupons were 102 mm x 102 mm (4 in x 4 in), which limited the length of friction stir processing that could be done on each plate. Friction stir processing was done using the Rockwell Scientific Company friction stir system under displacement control. Initial trials were conducted with a polycrystalline cubic boron nitride (PCBN) tool with a rotation rate of 400 RPM and a travel speed of 12.7 mm/min (0.5 in/min). The PCBN tool had a 24.1-mm (0.947-in) diameter shoulder and a 7.6-mm (0.30-in) diameter x 5.59-mm (0.220-in) long pin. The pin was slightly cone-shaped and contained three flats to promote material flow during friction stir processing. Additional trials were conducted using a tungsten-rhenium (W-Re) pin with a Densimet shoulder. This tool had a 24.1-mm (0.947-in) diameter shoulder and a 7.9-mm (0.3125-in) diameter x 5.84-mm (0.230-in) long, threaded pin. The processing conditions were a tool rotation rate of 600 RPM and travel speed of 12.7 mm/min. After processing, the microstructures of metallographically prepared and electropolished samples were documented using light microscopy and scanning electron microscopy (SEM). Grain size values were measured for various regions across the Nitinol plate.

The austenite and martensite transformation temperatures of the friction stir processed plate using the PCBN tools were measured using DSC. Samples ranging in mass from 24 to 28 mg were cut using a slow-speed diamond saw from the base metal and friction stir processed regions including the heat affected zone (HAZ), thermo-mechanical affected zone (TMAZ), and nugget. Heating and cooling cycles were repeated multiple times to ensure the accuracy of the data obtained from the DSC.

The friction stir processed plate using the W-Re tool was subjected to further processing to simulate the tube drawing process used for stent applications. This Nitinol plate was hot-rolled at 850°C from an initial thickness of 6.35 mm to a final thickness of 1.55 mm (0.061 in). The plate was rolled approximately 10% each pass (14 total passes) for a total reduction of 75.6%. The plate was reheated to the rolling temperature after each pass. Prior to rolling the FSP sheet, a test sheet of unprocessed, fully annealed Nitinol plate was hot rolled to 1.55 mm thickness. The microstructure of the FSP rolled sheet was documented with light microscopy and SEM.

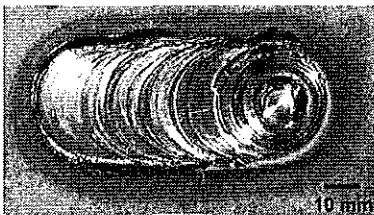
Transformation temperatures of the rolled sheet were measured using DSC. Fourteen 5-mm (0.2-in) diameter samples were laser cut across the width of the sheet encompassing rolled and FSP + rolled microstructures. The discs were then electropolished to remove the laser HAZ and surface oxides. DSC tests were done on each disc. The discs were then fully annealed at 850°C for 30 minutes, and the DSC tests were repeated – a common test procedure for Nitinol.

Tensile tests were conducted on the rolled and FSP + rolled microstructures. The samples were laser machined from the appropriate regions of the sheet following modified ASTM specification E8. Tensile tests were conducted with a cross-head displacement rate of 0.254 mm/min (0.01 in/min). Some Nitinol sheet samples were then deformed to 6% strain, unloaded, and then tensile tested to failure; a standard test for the superelastic properties of Nitinol.

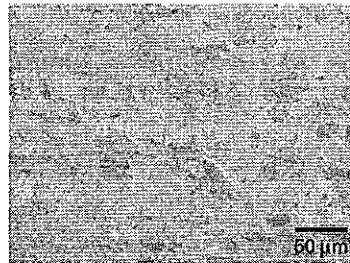
### Results and Discussion

A processed zone 76 mm (3 in) long was produced using the PCBN tool (Figure 3a). The processed region was free of macroscopic and microscopic defects (Figure 3b). No cover-gas was used during processing so some oxidation of the Nitinol surface occurred. A small volume of flash was produced during processing. The Nitinol did not produce any noticeable wear on the PCBN pin.

Grain size values across the processed Nitinol plate are shown in Figure 4. The average grain size in the center of the nugget region (Figure 3b) is 27  $\mu\text{m}$ . An initial grain size of approximately 45  $\mu\text{m}$  of the unprocessed Nitinol indicates some grain size refinement produced by friction stir processing. The greatest refinement occurred at the top of the nugget region where the shoulder and pin produce a large amount of plastic deformation. The grain size decrease in the processed regions is common in alloys that undergo friction stir welding and processing [13].



(a) Top View



(b) Microstructure of FSP nugget center

Figure 3. Void-free, defect-free friction stir processed region produced using the PCBN tool.

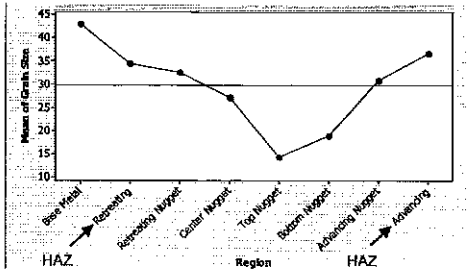


Figure 4. Grain size results for the friction stir processed Nitinol produced with the PCBN tool.

A typical output from a DSC measurement for FSP material is shown in Figure 5a. These results are fairly typical for Nitinol except for the fluctuation shown halfway through the austenite transformation (arrow, bottom curve). This effect was repeated in several DSC curves for the FSP material and may be due to an R-phase transformation. The variation in transformation temperature across the processed plate is shown in Figure 5b. The center of the nugget shows a lower transformation temperature than the surrounding material. This decrease in transformation temperature may be due to the chemical solutionizing effects and grain size refinement from friction stir processing.

A 95-mm (3.74-in) long processed zone was produced in the 6.35-mm thick Nitinol plate using the W-Re tool (Figure 6a). The morphology and microstructure are similar to that produced with the PCBN tool. The Nitinol did not produce any noticeable wear on the W-Re pin. The grain size of the processed nugget is similar to that measured for the PCBN-tool nugget. Transformation temperatures for this processed material were not measured because of the similarity of the microstructure compared to the PCBN tool processed zone.

The friction stir processed and hot-rolled sheet is shown in Figure 7. Friction stir processing did not affect the rolling characteristics of the sheet – it rolled exactly the same as an unprocessed sheet hot rolled as a test case. This is a clear benefit of the solid-state nature of friction stir processing. The microstructure of the rolled sheet indicated that some grain size refinement has occurred in the FSP region. Most of the cross section of the micrograph of Figure 7b shows of the refined grain size from the FSP nugget. Toward the bottom of the micrograph a coarser grain size is present, which was below the original processed zone before hot rolling.

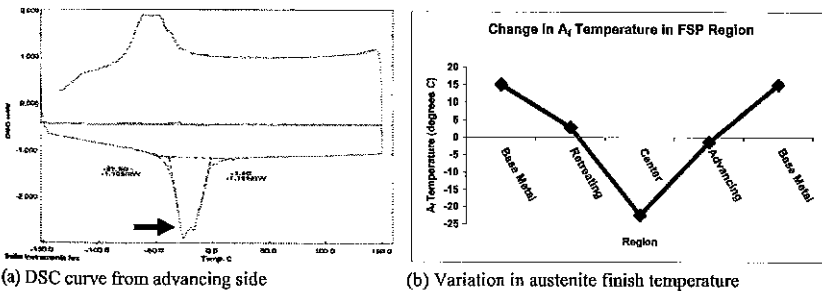
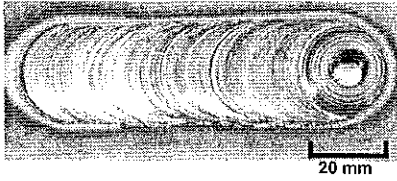
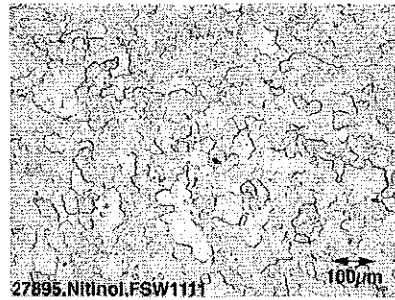


Figure 5. Transformation temperature results for the Nitinol friction stir processed with PCBN.

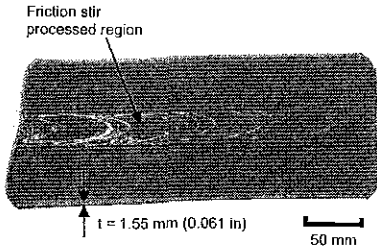


(a) Top View

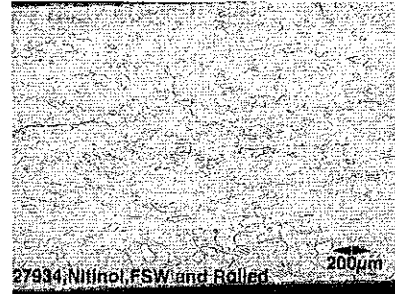


(b) Microstructure of FSP nugget center

Figure 6. Void-free, defect-free friction stir processed region produced using the W-Re tool.



(a) Overall View



(b) Microstructure of rolled FSP nugget

Figure 7. Friction stir processed and hot-rolled Nitinol sheet.

The DSC results for the rolled sheet are shown in Figure 8. A distance of around 5 cm corresponds to the center of the FSP nugget. These results are similar to those found for the PCBN tool FSP region with a reduction in the transformation temperatures in the center of the nugget. Tensile test results from the base metal and FSP regions (both hot-rolled) are presented in Figure 9. The tensile curves are typical of samples in the transverse orientation of rolled Nitinol sheet or drawn tubing [14]. These curves differ from those obtained from the longitudinal orientation of sheet, tubing, and wire samples in that there is not a distinct loading plateau in the 2 to 8% strain regions. These differences are due to texture effects, whereby the rolling direction contains  $[110]_{B2}$  and  $[111]_{B2}$  texture, whereas the transverse direction is dominated by the  $[100]_{B2}$  texture [15]. Nevertheless, these FSP tensile curves indicate that there are no brittle intermetallics ( $Ti_2Ni$ ) formed as in fusion-welded Nitinol [10, 11]. The higher strength in the FSP region may be because of the lower transformation temperature and finer grain size.

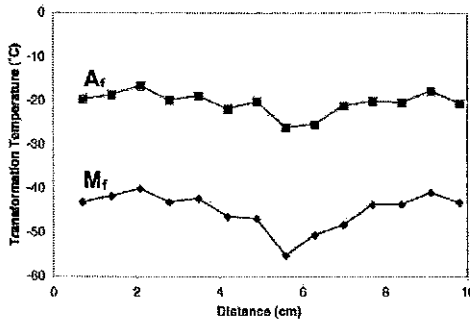


Figure 8. DSC results for the transformation temperatures of the FSP and hot-rolled Nitinol sheet.

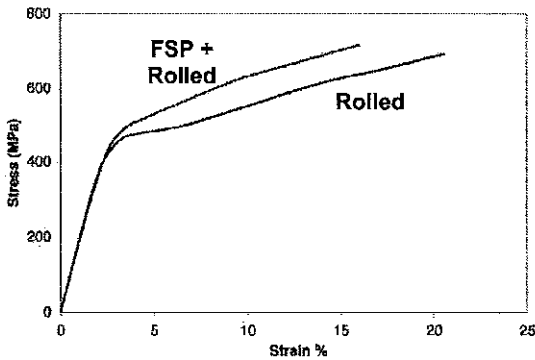


Figure 9. Tensile test results for the hot-rolled Nitinol sheet: base metal and FSP regions.

### Conclusions

Friction stir processing of Nitinol plate is feasible. The processing can be done with a polycrystalline cubic boron nitride or tungsten-rhenium tool. The Nitinol retains its shape memory and superelastic properties following processing. This was confirmed by differential scanning calorimetry of processed samples to determine their austenite and martensite transformation temperatures. A slight amount of grain size refinement in the friction stir processed zone caused an increase in strength and a decrease in the  $A_f$  transformation temperature compared to the base metal. Friction stir processed Nitinol can be hot rolled 76% at 850°C without cracking. These results point to a promising future process for friction stir welding of Nitinol alloys for medical device applications.

### Acknowledgements

This work was supported by Rockwell Scientific Company and Nitinol Devices & Components internal research and development funds.



## References

1. Wayman, C. M. and Duerig, T. "An Introduction to Martensite and Shape Memory", Engineering Aspects of Shape Memory Alloys, (Butterworth-Heinemann Ltd., London) 1990, 3:20.
2. Funakubo, H., ed., Shape Memory Alloys, (Gordon and Breach Science Publishers, New York) 1997.
3. Pelton, A. R., DiCello, J., and Miyazaki, S. "Optimisation of Processing and Properties of Medical-Grade Nitinol Wire", *Min. Invas. Ther. and Allied Technol.*, **9** (2000): 107-117.
4. Stöckel, D., Pelton, A. R., Duerig, T. "Self-Expanding Nitinol Stents: Material and Design Considerations", *Eur. Radiol.*, (2004) **14** 292-301.
5. Michal, G. M. "Diffusionless Transformations in TiNi", Ph.D. Thesis, Stanford University, 1979.
6. Otsuka, K., Kakeshita, T. "Science and Technology of Shape-Memory Alloys: New Developments.", *MRS Bulletin*, **27.2** (2002): 91-98.
7. Duerig, T., Pelton, A., Stöckel, D. "An Overview of Nitinol Medical Applications" *Materials Science and Engineering A* 273-275 (1999) 149.
8. *American Heart Association*. "Stent Procedure" <<http://www.americanheart.org/presenter.jhtml?identifier=4721>>.
9. Spectranetics. 2004. In-Stent Restenosis. <<http://www.spectranetics.com/cadt/brachy.html>>.
10. Harikawa, H., Ueki, T., and Shiroyama, K., "Superelastic Performance of Ni-Ti Thin Tubes", *SMST-94 Proceedings of the International Conference on Shape Memory and Superelastic Technologies*, 1994, 347-352.
11. Schlossmacher, P., Haas, T., and Schuessler, A., "Laser Welding of NiTi Shape Memory Alloys", *SMST-94 Proceedings of the International Conference on Shape Memory and Superelastic Technologies*, 1994, 85-90.
12. Hodgson, D. "Fabrication, Heat Treatment and Joining of Nitinol Components." *SMST- 2000 Proceedings of the International Conference on Shape Memory and Superelastic Technologies*, (2001): 11-24.
13. Mishra, R.S. "Friction Stir Processing Technologies." *Advanced Materials and Processes*, **161** (10) (2003): 43-46.
14. Gong, X-Y, Pelton, A. R., and Duerig, T. W., "Orientation Effects on the Mechanical Properties of Nitinol Tubing", submitted to SMST-2004.
15. Robertson, S. W., et al., "Texture in Tubes and Plates of Nitinol", *SMST- 2003 Proceedings of the International Conference on Shape Memory and Superelastic Technologies*, (2004): 341-347.