Shape memory effect, temperature distribution and mechanical properties of friction stir welded nitinol

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ABSTRACT

Welding of shape memory alloys without deterioration of shape memory effect could vastly extend their applications. To retain shape memory behavior, a solid-state welding technique called friction stir welding was employed in this study. Austenitic NiTi alloy sheets of thickness 1.2 mm were joined at tool rotational speeds of 800, 1000, and 1200 rpm. Due to dynamic recrystallization, the grain refinement has occurred in the weld region. The tensile testing has shown superelastic plateau for the welds at 800 and 1000 rpm. The phase transformation behavior of different weld regions was studied in detail using differential scanning calorimeter. A marginal drift in transformation temperatures was observed in the weld. To understand the drift in phase transformation temperatures, finite element analysis was carried out with focus on temperature distribution during welding. Finally, time-dependent shape recovery of a FSW welded joint was studied and it was found that the original position was completely recovered after 27 s at a temperature of 65 °C.

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1. Introduction

Nickel-Titanium or Nitinol alloys are widely used shape memory alloys (SMA) due to their exhibition of excellent thermomechanical behavior, pseudoelasticity and biocompatibility. They have been explored in various engineering fields as actuators, sensors, structural elements [1,2] and also as braced frames for seismic retrofitting of civil structures [3]. The applicability of these alloys is limited due to its low formability. Hence, the manufacturing of complex shapes tends to be difficult and expensive. Therefore, the welding becomes inevitable to fabricate NiTi SMA into different geometrical structures with adequate freedom of design. Many conventional and modern fusion welding processes such as gas tungsten arc welding [4,5], plasma welding [6], brazing [7], resistance welding [8], laser welding [9,10] and electron beam welding [11] have been

used to weld NiTi. Since NiTi SMA is highly sensitive to microstructural and compositional changes, the fusion welding of these alloys leads to deterioration of the mechanical and shape memory properties [12,13]. In fusion welding, due to melting and rapid cooling, dendrite columnar microstructure is formed along with brittle intermetallics [14]. These changes during welding drastically alter the transformational temperatures and reduce the thermo-mechanical stability of the weld joint.

Recently, we have demonstrated the friction stir welding (FSW) of NiTi alloy with good mechanical properties and retention of shape memory behavior with a minimal change in transformational temperatures [15]. In friction stir welding, the rotating tool having a pin and a shoulder will plunge into the base material and welds it together [16,17]. FSW process produces autogenous weld and the issue of compositional compatibility of filler material with base metal can be ignored [18]. Due to solid state conditions, the distortion and residual stresses will also be minimal compared to fusion welding processes.

During FSW at particular rotational speed, the temperature and

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strain gradient exists across the weld which results in regions such as nugget or stirred zone, advancing side and retreating side [19]. The tool rotational speed is the most influential parameter of the FSW process [18,20]. Recently, the influence of tool rotational speed on microstructure and mechanical properties has been reported for materials such as Al-Li alloy and steel [21,22]. As the tool rotation speed increases, the heat induced due to friction between the tool-workpiece interface and the plastic strain energy from intense deformation of workpiece increases. Hence, the tool rotating at higher speed will generate more heat and hence reach higher peak temperature. In addition to the effect on peak temperature, the tool rotational speeds also have impact on strain and strain rate.

As tool rotational speed is major parameter, in this study an attempt has been made to weld NiTi shape memory alloy at three different rotational speeds. The effect of tool rotational speed on the microstructure, phases and mechanical properties of the NiTi alloy has been studied. The phase transformation behavior with respect to the rotational speeds and in different weld regions has been explored. Further, in order to understand the temperature distribution across the weld, the FSW of Nitinol was simulated using finite element method (FEM). The shape memory effect of the welded plate was studied using hot plate actuation technique.

2. Experimental details

The austenitic NiTi sheet of 1.2 mm thickness with Ni 50.75 at.% (SE508-NDC) was used for welding. The sheets were cut into dimensions 50 mm × 70 mm and welded using five-axis FSW machine (BiSS-ITW Bangalore) in closed square butt configuration.

Experiments were conducted at three rotational speeds (800 rpm, 1000 rpm and 1200 rpm) with constant traverse speed of 50 mm/min in the ambient environment without any cooling medium or shielding gas. A tungsten alloy (densimet) tool was used for welding. Microstructure, mechanical and shape memory properties of the welds were characterized. Fig. 1 shows the schematic of FSW and process parameters used for welding.

The samples for microstructural analysis were polished using various grits of SiC paper, then using alumina powder and finally etched using 50 mL H₂O + 40 mL HNO₃ + 10 mL HF solution. The microstructures were recorded using Carl Zeiss inverted optical

microscope. The phase analysis was carried out by X-ray diffraction (XRD) technique using X-Pert PRO PANalytical. Further, tensile test samples with a cross section of 1.2 mm × 4 mm were machined across the weld. Tensile test was carried out on Instron-5967 machine at a strain rate of 10^{−3}. Vickers microhardness measurements were taken along the cross section of the weld using the FM-800 Future Tech Corp at 500gf load for 15 s. The characterization techniques to evaluate the microstructures, phases, hardness and stress-strain behavior were performed at room temperature.

Samples were cut from nugget, retreating side and advancing side of the weld joint (Fig. 2) for studying the phase transformation behavior using differential scanning calorimetry (DSC). The phase transformation behavior was studied using Netzsch-DSC214 Polyma DSC machine at a heating/cooling rate of 10 °C/min with a constant flow of nitrogen.

The FSW of Nitinol has been simulated using Comsol Multiphysics 5.3a based on FEM. To study the shape memory effect, the welded samples were annealed at 475 °C for 1 h and then furnace cooled to room temperature. A strip of dimension 65 mm × 2 mm × 1.2 mm was cut across the weld and used for actuation studies.

Fig. 2. Schematic of the DSC sample regions (A-Nugget, B-Advancing side and C-Retreating side).

Fig. 1. FSW process and the process parameters used for welding.

3. Theoretical analysis

The model used for the study was based on the work done by Song et al. [23]. The heat input from both the tool pin and shoulder was considered. The governing equation of heat generation in pin-workpiece (Q_{pin}) considering the evolution of heat due to shearing of the workpiece and friction is given by,

$$Q_{pin} = \frac{2\pi n \mu}{\sqrt{3(1 + \mu^2)}} \bar{Y} R_p \quad (1)$$

The heat generation at the shoulder-workpiece ($Q_{shoulder}$) interface is given by,

$$Q_{shoulder} = 2\pi \mu \frac{F_n}{A_{shl}} R_s n \quad (2)$$

Where μ -coefficient of friction, R_s -shoulder radius (mm), F_n -plunge force (KN), A_{shl} -surface area (mm^2), n -tool rotational speed (RPM), $\bar{Y}$ -average shear stress of the material and R_p -pin radius (mm).

On heat generation due to the rotation of the tool, the heat will transfer from the vicinity of the weld to the surrounding via both convective and radiative mode of heat transfer. The governing heat transfer equations for conductive heat transfer in the material (Q) and convective-radiative heat transfer from the workpiece is given by the following equations.

$$\rho C_p u \bullet \nabla T = \nabla \bullet (k \nabla T) + Q \quad (3)$$

$$Q = hA(T_0 - T) \quad (4)$$

$$Q = \varepsilon \sigma (T_0^4 - T^4) \quad (5)$$

Where ρ -density (Kg/m^3), C_p -specific heat ($J/Kg K$), u -welding speed (mm/min), k -thermal conductivity ($W/m K$), h -heat transfer coefficient ($W/m^2 K$), A -surface area of exposed surface (mm^2), ε -surface emissivity, σ -Stefan-Boltzmann constant and T_0 -ambient temperature.

4. Results and discussion

4.1. FSW and microstructural evaluation

The macroscopic images of the welds produced at 800, 1000 and 1200 rpm are shown in Fig. 3a–c. All the welds had proper consolidation of the joint without any surface defects. The macro-structure of the weld cross section was shown in Fig. 3d. The zone A-B represents the transition zone from shoulder region to base metal on advancing side and retreating side of the weld respectively.

The microstructures of the base metal and the weld are shown in Fig. 4. The base metal has equiaxed coarse grains. The micro-structure of the weld region for all the tool rotational speeds has fine recrystallized grains. The recrystallized grains were found throughout the weld and there was no clear distinction between nugget, thermo-mechanically affected zone (TMAZ) and heat affected zone (HAZ). Generally, the dynamic recrystallization takes place in the metals when subjected to hot working and it depends upon the peak temperature, plastic strain, strain rate and stacking energy of the materials [24]. In metals with high stacking fault energy like NiTi, the continuous dynamic recrystallization occurs when the temperature is greater than 50% of the melting point [25,26]. During FSW, the frictional heat and plastic deformation caused by the tool rotation induces the welding temperature of approximately 0.7–0.8 times the melting point of the material [27]. Hence, the continuous dynamic recrystallization accompanied with dynamic recovery has resulted in the formation of refined elongated grains during FSW of NiTi alloy [20]. In the weld at particular tool rotational speed, the temperature and plastic deformation in the adjacent regions to the nugget are lower than the nugget zone. Due to this, the recrystallization does not take place outside the nugget for most of the materials. However, in NiTi alloy, even at lower strain and higher temperature recrystallization will occur [28]. This has resulted in the formation of recrystallized grains in TMAZ and HAZ and all the regions exhibited homogeneous microstructure.

Fig. 3. Defect free friction stir welds at a) 800 rpm b) 1000 rpm c) 1200 rpm and d) Macrostructure of the weld cross section.

Fig. 4. Microstructures of the NiTi alloy before and after welding a) Base metal, b) weld nugget at 800 rpm, c) weld nugget at 1000 rpm and d) weld nugget at 1200 rpm.

Fig. 5. XRD patterns of NiTi alloy before and after welding a) Base metal, b) FSW at 800 rpm, c) FSW at 1000 rpm and d) FSW at 1200 rpm.

4.2. XRD analysis

The XRD patterns of the base metal and weld regions at different rotational speeds are shown in Fig. 5. The base metal and the weld at three different tool rotational speeds have austenite (B2-cubic) phase with traces of martensite (B19'-monoclinic) phase. Inter-metallic compounds (NiTi_2 and TiNi_3) commonly observed in fusion welding of NiTi and are detrimental to shape memory behavior were absent. Further, interestingly phases of the base metal have been retained even after welding. However, in case of electron beam and laser beam welding, transformation between the martensite and austenite phases after welding was generally observed [11,29].

4.3. DSC analysis

The DSC curves of the base metal and the weld nuggets at different tool rotational speed are shown in Fig. 6. A_s , A_f , M_s and M_f are the austenite-martensite start and finish transformation temperatures. These transformation temperatures are given as an inset in Fig. 6. The DSC curves of base metal and weld nuggets at 800 and 1000 rpm depict the exothermic peaks during the forward transition from austenite to martensite ($B2 \rightarrow R \rightarrow B19'$ or $B2 \rightarrow B19'$) on cooling. Further, they depict the endothermic peaks during the reverse transition from martensite to austenite ($B19' \rightarrow B2$) on

heating. Fig. 6d shows the DSC curve of the weld nugget at 1200 rpm during heating and cooling cycles. The transformational temperatures of the base metal and weld nugget of 800 rpm were very close to each other. However, the martensite transformation temperatures (M_s and M_f) of weld nuggets of 1000 have increased after welding. Figs. 7 and 8 shows the DSC curves of advancing and retreating side of the weld at different tool rotational speeds. The phase transformation temperatures of both sides of weld at 800 rpm were close to the base metal. But the martensite transformation of both sides of the weld at 1000 rpm has shown a drift towards the positive direction. In case of the weld at 1200 rpm, the transformations are different and unusual from the base metal and the other welds. So, these transformations are represented as T_s and T_f during heating and cooling cycles respectively. This unusual pattern could plausibly due to microstructural heterogeneities and tool inclusions across the weld regions.

FSW is a thermo-mechanical process which involves high temperature and severe plastic deformation. As it was seen from the microstructures (Fig. 4) of welds at different tool rotational speeds, grain size of the nugget varied considerably. Further, it can be assumed that the dislocation density and the residual stress of the weld at a particular tool rotational speed will be different. As phase transformational behavior is sensitive to dislocation density, grain size and residual stress, the friction stir welded NiTi has exhibited a drift in phase transformational temperatures [30]. Due

Fig. 6. DSC curves of NiTi alloy before and after welding a) Base metal, b) Nugget of the weld at 800 rpm, c) Nugget of the weld at 1000 rpm and d) Nugget of the weld at 1200 rpm.

to high plastic deformation in the nugget zone, the temperature rise is comparably higher than the advancing and retreating sides [18,20]. However, the zones next to the nugget i.e. both TMAZ and HAZ experiences low to negligible plastic deformation and lower temperatures than the nugget zone. Hence during FSW, an inherent asymmetry in temperature, strain and strain rate exists across the weld. Typically, the advancing side encounters higher temperature and shearing rates than the retreating side [19]. This asymmetry in the temperature and plastic deformation across the weld regions may cause difference in dislocation density and residual stress which has led to change in phase transformation temperatures in different weld regions [28].

The hysteresis due to dissipative work is the interval between heating and cooling transformations i.e. $|A_s - M_f|$ and $|M_s - A_f|$ [31–33]. The hysteresis for the base metal and the weld at different rotational speed is given in Table 1. The hysteresis ($A_s - M_f$) has reduced for weld at 1000 rpm whereas increased for the weld produced at 800 rpm.

4.4. Microhardness and tensile analysis

Microhardness of the weld cross section and scanning electron microscope (SEM) images of the weld at 1200 rpm is shown in Fig. 9. The average hardness of the weld at 800 rpm, 1000 rpm and 1200 rpm were 275 HV, 262 HV and 284 HV respectively. Few abnormally high hardness values were noticed in 1200 rpm weld due to tool fragments (Fig. 9b) included and distributed across the

weld region. The distribution of tool fragments across the weld could be attributed to the severe plastic deformation and high temperature experienced at 1200 rpm than the other rotational speeds. The weld at 800 rpm and 1000 rpm has showed marginally lower hardness than the base metal (BM).

Fig. 10 shows the engineering stress-strain curve of the base metal and the welds. The weld produced at 800 rpm has displayed an ultimate tensile strength of 492 MPa and yield strength of 453 MPa. Interestingly, the weld produced at 1000 rpm had ultimate tensile strength of 638 MPa (66% of the BM) and yield strength of 602 MPa which is 17% higher than the base metal. The weld at 1000 rpm has a crucial superelastic plateau near 600 MPa until 7.25% strain while the weld at 800 rpm showed close to 460 MPa until 5% strain. Ultimate strength of the weld at 1200 rpm was 345 MPa which was lower among the welds produced due to the presence of hard tool fragments. The cross section of the weld (Fig. 10b), shows cracks close to the tool fragments which could be attributed to inhomogeneous material flow and mismatch in thermal expansion between the tool fragments and the NiTi.

In case of welding of shape memory alloys, the retainment of crucial superelastic plateau (functional property), yield strength and ultimate tensile strength are the factors defining the quality of the weld. The FSW of nitinol has shown better yield strength (due to grain refinement) than base metal at 1000 rpm and the welds at 800 and 1000 rpm has shown crucial superelastic plateau. It is important that the FSW has shown higher yield strength (compared to base metal) than most of the reported welding

Fig. 7. DSC curves of advancing side of friction stir welded NiTi alloy a) FSW at 800 rpm, b) FSW at 1000 rpm and c) FSW at 1200 rpm.

Fig. 8. DSC curves of retreating side of friction stir welded NiTi alloy a) FSW at 800 rpm, b) FSW at 1000 rpm and c) FSW at 1200 rpm.

Table 1
Hysteresis of the phase transformation behavior at different regions of the weld.

Weld region	Process conditions	$ A_s - M_f $ (°C)
Nugget	Base metal	56
	800 rpm	64
	1000 rpm	46
	1200 rpm	—
Advancing side	800 rpm	61
	1000 rpm	58
	1200 rpm	—
Retreating side	800 rpm	59
	1000 rpm	43
	1200 rpm	—

techniques such as electron beam welding, laser welding, tungsten arc welding [4,34–36]. On the other hand, the ultimate tensile strength depends on the bonding of the weld joint and the weak point across the weld structure acts as the nucleating point for fracture. In FSW of nitinol, the interface between the base metal and the weld region (transition zone from refined grains of weld to coarse grains of base metal) acts as weak point for the fracture. All the welded samples have broken at heat affected zone i.e. the weak interface zone. So, the joint efficiency can be improved by further detailed optimization on tool selection, tool geometry design and

process parameters to enhance the plasticization of material across the weld zones.

The fracture surfaces of the weld and the BM observed using scanning electron microscope are shown in Fig. 11. The fracture surface of the BM and the welds except at 1200 rpm showed dimples indicating ductile fracture. The fracture surface of the weld at 1200 rpm show smooth surface indicative of brittle fracture. Further, the micro cracks which could have been originated at the tool fragments are seen in Fig. 11d.

4.5. Shape memory effect

Since the weld at 1000 rpm showed better mechanical properties, it has been used for actuation studies. The welded sample was annealed at 475 °C for one hour before the actuation studies. The phase transformation behavior after annealing is shown in Fig. 12. Annealing has converted the welded NiTi from superelastic state to shape memory state i.e. from austenite to martensite at room temperature. Strain was imposed in the welded sample to bend to a U shape in an ice bath. After bending, the sample was actuated on a hot plate held at 65 °C. This temperature was chosen as it was above the austenite finish temperature of the sample. The sample was not disturbed until imposed strain was completely recovered. The actuation was quantified using recovery angle, which is the angle between two tangents drawn 5 mm from the weld center on either

Fig. 9. a) Microhardness of the weld cross section and b) Cross sectional SEM images of the weld at 1200 rpm showing tool fragments.

side. The recovery angle was measured using the AutoCAD software from the images acquired during actuation. The method of angle measurement was similar to the method reported by J. P. Oliveira [37]. The duration for complete strain recovery was noted to be 27 s, beyond which there was no change in the sample. The load applied for bending will convert the sample from twinned martensite into detwinned martensite. On heating, austenite transformation starts and the sample will return to initial straight position. The schematic of angle measurement, recovery angle vs time plot and snapshots of the actuation are shown in Fig. 13.

Fig. 10. a) Engineering stress-strain plot of the base metal, b) Engineering stress-strain plot of the welds and c) Cross sectional SEM images of the weld at 1200 rpm showing cracks.

Fig. 11. Fracture surfaces of the tensile specimen a) Base metal, b) weld at 800 rpm, c) weld at 1000 rpm and d) weld at 1200 rpm.

Fig. 12. DSC curve of the annealed sample after welding.

4.6. Simulation results based on FEM

The governing equations mentioned in theoretical analysis section have been used for simulation. Based on the same model, the temperature distribution during FSW of stainless steel and aluminum was reported [38,39]. The welding parameters

mentioned in Fig. 1 and the material properties of tool and work piece has been used for simulation. The temperature distribution at different tool rotational speed is shown in Fig. 14a–c. The increase in tool rotation speed has increased the maximum temperature during welding. The FSW at 1200 rpm has shown a highest temperature of 1094 °C. The maximum temperature was recorded in the edge of the pin on the advancing side. The advancing side has shown higher temperature than the retreating side for all the tool rotational speeds. The temperature of the weld at different points across the weld viz. nugget center (NC), peak temperature (PT), advancing side (AS) and retreating side (RS) is shown in Fig. 14d and e. The temperature data from the simulation further corroborates the DSC results and microstructural results i.e. with increasing rotational speed the material is subjected to higher temperatures resulting coarsening of grains leading to greater drift in transformational temperatures. Further, drift in transformational temperatures within a weld can be attributed to variation in temperature experienced by each region.

5. Conclusions

The FSW of NiTi SMA at three different rotational speeds has been carried out and the following conclusions are drawn.

FSW is a feasible method to join SMA as the weld has formed without intermetallics and the shape memory behavior was retained after welding.

Fig. 13. a) schematic of angle measurement, b) recovery angle vs time plot and c)-f) Snapshots during actuation.

Recrystallized grain size in the nugget has increased with the increasing rotational speed due to considerable rise in peak temperature.

The phase transformation temperatures of the weld produced at 800 rpm and base metal were near identical to the base metal. However, the welds at 1000 and 1200 rpm have shown a marginal drift from the transformation temperatures of the base metal as they were significantly affected by high temperature during processing.

The asymmetry of weld condition across the weld has led to drift in transformation temperatures in nugget, advancing, and retreating sides.

The weld accomplished at 1000 rpm has showed higher yield strength than the base metal and crucial superelastic plateau near 600 MPa.

The weld joint efficiency at 800 rpm and 1200 rpm was low and further optimization of welding parameters is required for improving the weld structural integrity.

Fig. 14. Temperature distribution during FSW of Nitinol a) Weld at 800 rpm b) Weld at 1000 rpm c) Weld at 1200 rpm, d) Schematic of temperature probe and e) Comparison plot.

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