Journal of Materials Science: Materials in Engineering

ORIGINAL PAPER Open Access

Enhancing Ni–Ti shape memory alloy diffusion bonding with Ti/Ni reactive multilayer foils

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Abstract

This study investigates the utilization of Ti/Ni reactive multilayer foils as an energy source for facilitating the joining of Ni–Ti shape memory alloys through diffusion bonding. Multilayered samples were prepared using a 10-cycle accumulative roll bonding (ARB) process to be used for the bonding process. Diffusion bonding employing reactive multilayers was conducted over a temperature range of 600 °C to 900 °C, at 5 MPa pressure, with a 1-h hold time. Additionally, a comparison was made with a diffusion-bonded Nitinol sample at 900 °C without a reactive multilayer. Materials characterization and testing involved scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), shear strength testing, and differential scanning calorimetry (DSC), which were conducted on the bonded samples. The findings underscored the advantages of using reactive multilayers for diffusion bonding. These benefits included the formation of TiNi and the induction of a shape memory effect in the joint region, alongside a 1.5 times shear strength compared to identical diffusion bonding conditions without reactive multilayers. Moreover, employing reactive multilayers in the diffusion bonding of Nitinol holds promise for significantly reducing the energy needed to achieve robust and seamless bonded boundaries in the joining area.

Keywords Accumulative roll-bonding, Diffusion bonding, Ti/Ni reactive multilayers, Nitinol

Introduction

Nowadays, the demand for Nitinol in the industry is increasing significantly due to its remarkable properties, including shape memory effect and superelasticity. Nitinol is used in numerous industries, especially in healthcare, where it is valued for its excellent biocompatibility (Falvo et al. 2005). Given the low formability of intermetallic compound alloys, welding and bonding are critical manufacturing methods for producing complex-shaped parts with these materials. Various welding methods have been reported for joining final products made from Ni–Ti alloys, including electric arc welding (Eijk et al. 2003; Ikai et al. 1996), laser beam welding (Pfeifer, et al. 2006; Dong et al. 2006), soldering (Zhao et al. 2009), adhesive bonding (Rossi et al. 2008; Man and Zhao 2006), friction welding, and diffusion bonding (Salazar et al. 1997; Kejanli et al. 2009; Emadinia et al. 2015). It should be noted that the shape memory properties of welded joints generally differ fundamentally from those of the base metals. Therefore, selecting an appropriate joining method that minimally impacts mechanical and chemical properties, such as diffusion bonding, is essential to achieve a proper final welded product (Oliveira et al. 2017). Diffusion bonding methods (Salazar et al. 1997) have the advantage of producing perfect joints without filler materials, but they often require high temperatures and long holding times. To overcome these challenges, various techniques have

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been developed, such as the use of higher pressure or reactive multilayers (Emadinia et al. 2015; Lin et al. 2012), to improve the conditions for diffusion bonding of Ti-alloys, thus avoiding the need for high temperatures or long holding times.

The utilization of reactive multilayers in diffusion bonding can enhance joint conditions and improve the mechanical properties of joints. In addition, some research has been proposed the use of reactive multilayer films for bonding metals to ceramics as an effective method for joining (Lin et al. 2012; Qiu and Wang 2008). The advantages of this method include the elimination of the need for a furnace due to the high speed of the exothermic reaction and the concentrated heat. It is worth noting that many different foils have been produced, but only a limited number, including Ni/Al and Al/Ti, have been used for bonding solely due to their sufficient exothermic reaction enthalpy and high adiabatic reaction temperature (Barin et al. 1977; Weihs 2014). Compared to the previously discussed types of foils, Ti/Ni reactive multilayer foils are less frequently used for bonding alone due to their lower reaction enthalpy. Adams et al. (Adams 2015) reported an exothermic reaction rate of 0.1 to 1.4 m/s for Ti/Ni reactive multilayer foils. Another source has highlighted the formation of phases such as NiTi martensite with a monoclinic B19′ structure, TiNi2 with a hexagonal structure and NiTi with a B2 structure after the reaction of the Ti/Ni reactive multilayer (Adams, et al. 2009). Additionally, Lenret et al. (Lehnert et al. 2000) demonstrated that the shape memory effect results from the formation of the B19′ structure.

As mentioned above, these films have not found widespread application in stand-alone bonding due to the low reaction enthalpy of reactive Ti/Ni multilayer films. Therefore, Cavalro (Cavaleiro et al. 2014a, 2014b) proposed the application of Ti/Ni reactive multilayer foils for diffusion bonding of Ni–Ti and Ti6Al4V alloys. Others explored the use of reactive multilayer foils for the bonding of various alloys, such as the Ti–Al alloy. For instance, Kavo et al. (Cao et al. 2008) showed that the use of an Al/Ni reactive multilayer foil for the diffusion bonding of Ti-48Al-2Cr-2Nb alloy (Cao et al. 2008) significantly reduced the temperature and bonding conditions compared to diffusion bonding of Ti-47Al-4/5 alloy (Cr, Mn, Nb, Si, B) (Çam et al. 1997) (without reactive layers). This reduction in temperature and bonding time represents a decisive advantage of using reactive multilayer foils in bonding processes. Finally, Emadinia (Emadinia 2013) investigated the use of cold-rolled Ti/Ni reactive foil for the diffusion bonding of Ti6Al4V alloy with a Ni–Ti memory alloy. The study found a relatively good microstructural match between the two Ti alloys, which served as base metals, and the Ti/Ni reactive multilayer foil used to facilitate the bonding of the components (Emadinia et al. 2015).

While numerous studies have investigated various aspects of bimetal multilayers (Adams 2015), and research on the utilization of Ti/Ni multilayers produced using Accumulative Roll Bonding (ARB) for bonding applications (Emadinia et al. 2015) has rarely been reported. Specifically, the use of Ti/Ni multilayers produced by cold ARB to enhance the diffusion bonding of two pieces of Nitinol has not been thoroughly explored. Therefore, this study aims to produce Ti/Ni multilayer materials with a substantial number of layers, up to approximately 7000 layers, using ARB over 10 cycles without inter-pass heat treatment. Subsequently, the study will examine the mechanical properties and shape memory behavior of the diffusion-bonded regions, along with conducting microstructural studies, to demonstrate the improvement in bonding conditions achieved with the use of reactive multilayers, which eliminate the need for high temperatures and long holding times, thus contributing to energy savings.

Experimental procedure

Evaluation of the base metal

The microstructure of the Nitinol sheet (nickel-titanium shape memory alloy) was observed using an optical microscope. For this purpose, a solution of 1 part HF, 5 parts HNO_3, and 5 parts CH_3COOH was used for etching for 10 s after surface preparation.

In order to determine the mechanical properties of the base metal, tensile and hardness tests were carried out on a 0.75-mm thick Nitinol sheet. The standard flat Nitinol tensile was stretched at ambient temperature using a Santam SAF-50 automatic single-axis tensile machine. The final tensile strength of the Nitinol sheet was determined based on the maximum force and the initial cross-sectional area of the sample.

In addition, to determine the transformation temperatures of the base metal, a disk-shaped sample with a diameter of 5 mm was cut from the Nitinol sheet, and a calorimeter was used to measure the heat. The test was performed according to the ASTM D 3418–1 standard with a cooling and heating rate of 10 °C/min in a nitrogen environment.

Diffusion bonding of Nitinol with and without Ti/Ni reactive multilayer

In the experimental phase of this study, we performed diffusion bonding of the base metal (Nitinol) both with and without the use of a Ti/Ni reactive multilayer. Subsequently, the bonded samples were subjected to a thorough examination to evaluate their mechanical, microstructural, and shape memory properties, focusing

on the bonding area. Nitinol (52.62 at. % Ni, 0.067% at. % C) plates with a thickness of 0.75 mm, which are intended for medical purposes and are manufactured by the Kloss Research Laboratory, were used as the base metal. According to the manufacturer's specifications, this alloy has a martensitic structure at low temperatures. Consequently, it can be deformed at zero °C and when placed in the hand, the shape reverses due to its transformation temperature, which is around 30 °C.

The production of bimetal Ti/Ni lamellar composites involved accumulative rolled bonding (ARB) of commercial pure Ni and Ti foils, with thicknesses of 150 and 200 µm, respectively. This process was carried out without any heat treatment according to Fig. 1. Alternatively, four Ti and three Ni foils were stacked to create a 1.25-mm initial sandwich with approximately 36 vol.% of Ni. To prepare the initial metallic multilayer composites, Ni and Ti foils were degreased in acetone for 30 min, scratch-brushed, and alternately stacked. The stack, fixed with copper wires, was rolled at room temperature to reduce thickness, then cut, degreased, brushed, stacked, fixed, and roll-bonded again. This process was repeated up to 10 times without lubrication or heat treatment, ensuring at least 50% thickness reduction per cycle. Surface treatment and roll-bonding were performed within 60 s to prevent oxide layer formation. Detailed information on the ARB production method can be found in our previous study (Mokhles et al. 2020). The rolling processes were carried out consecutively without pre-annealing treatment to produce 10 cycles of accumulative roll bonding (ARB)-processed multilayers, intended for application in the diffusion bonding of Nitinol. Our selection of the 10-cycle ARB-processed multilayers was informed by the results of a previous thermal study, where annealing was performed on multilayers produced from 2, 6, 8, and 10 cycles of ARB processing. Among these, the 10-cycle ARB-processed multilayers exhibited optimal thermal properties, characterized by the lowest activation temperature and significant activation energy, as determined in our prior investigation (Mokhles et al. 2024).

Based on the research process, the diffusion bonding of Nitinol without using multilayer as a reference was investigated in this section. For this purpose, two Nitinol strips with dimensions of $0.7\times70\times5$ mm^3 were used for the diffusion bonding process. To ensure optimal bonding conditions, a 5×5-mm^2 area on each strip was polished to a high gloss and smoothness with a 1-µm diamond suspension during surface preparation. Diffusion bonding experiments were carried out in a YARAN Torr 3^{-10} vacuum furnace at 900 °C, applying a pressure of 5 MPa, with a holding time of 60 min. The heating speed of the furnace was set at 10 °C/min to facilitate the bonding process. Subsequently, the effect of the presence of Ti/Ni reactive multilayers in the diffusion bonding of Nitinol samples was investigated by experimental analysis. The conditions for surface preparation of the samples remained the same as before, with the surface of the Ni/Ti reactant completely smooth and polished. Figure 2a illustrates an example of the surface preparation of a shape memory alloy and a Ti/Ni reactive multilayer used in diffusion bonding. As can be seen in Fig. 2b and c, the samples were loaded with a 310 SS fixture. The desired clamping force was achieved by tightening two M10 bolts and nuts with a calibrated torque wrench. The conditions for diffusion bonding, including the applied force, holding time, and heating rate, were quite similar to those in the reference condition. Only the holding temperatures were different, and the temperatures chosen were 900, 800, 700, and 600 °C, all of which are higher than the activation temperature of the 10-cycle ARBed Ti/Ni reactive multilayer (500 °C) (Mokhles et al. 2024).

The furnace used for the diffusion bonding process, as shown in Fig. 2d, played a critical role in controlling

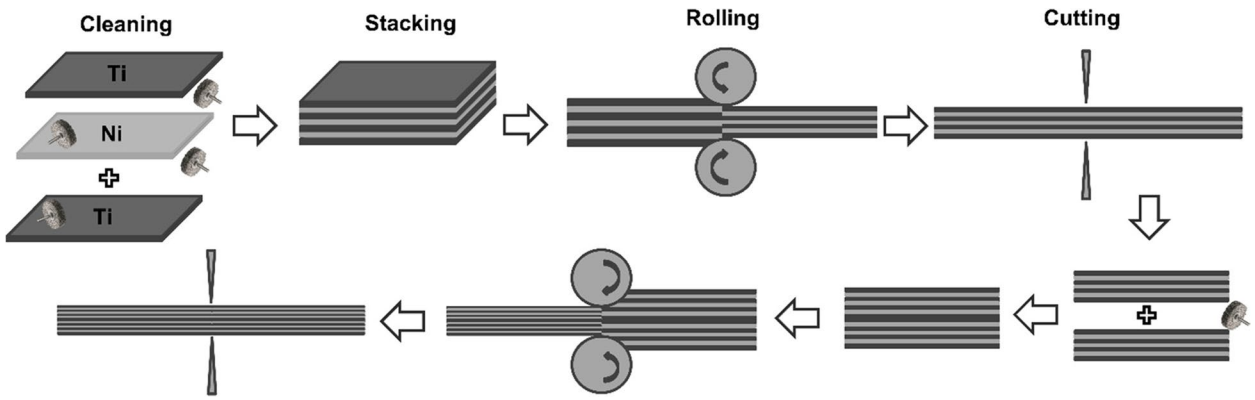


Fig. 1 Schematic of accumulative roll bonding process used for producing Ti/Ni reactive multilayers

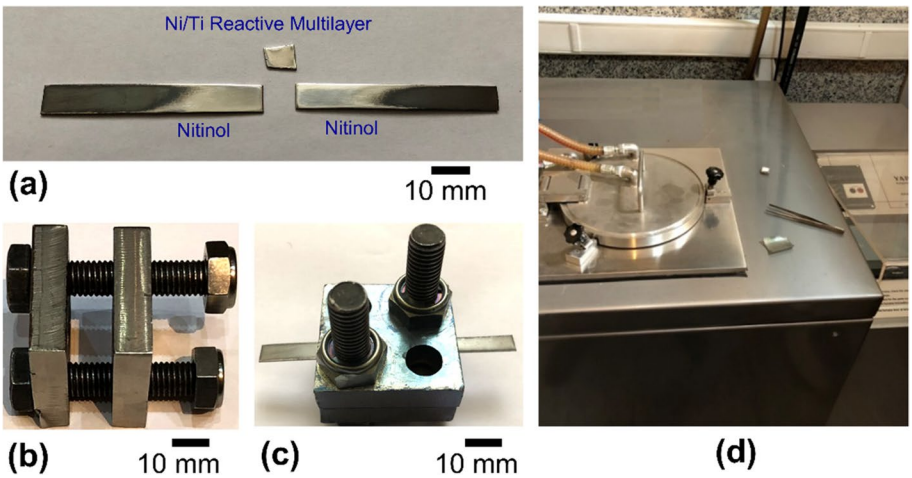


Fig. 2 **a** Surface preparation for diffusion bonding, **b**, **c** Clamping fixture used for applying load force during the bonding process, and **d** YARAN furnace used for the diffusion bonding

the bonding temperatures and ensuring uniform heating throughout the samples.

Characterization of the bonded joints

The microstructural and chemical composition of the composite area of the prepared samples and the base metal was analyzed using a high-resolution scanning electron microscope (FESEM) at 15 kV and an EDS spectrometer calibrated with Linked ISI software.

To evaluate the shear strength of the lap joints in the prepared samples, the SANTAM SAF-50 machine was used at a strain rate of 0.1 mm/min at room temperature. After the test, the actual contact area of the two samples was measured and used to calculate the shear strength by dividing the maximum force by the actual fracture area.

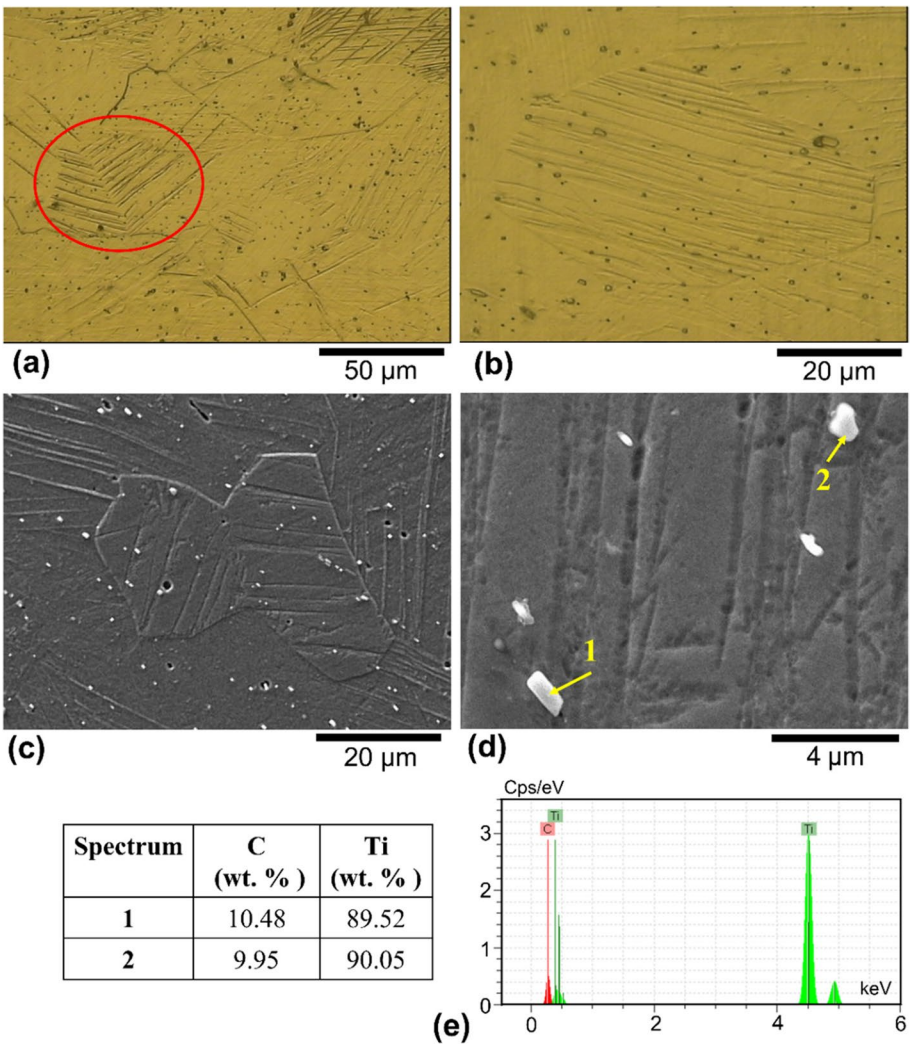
Finally, to determine the transformation temperatures and evaluate the shape memory effect of the bonding area, 5 mm diameter disc-shaped samples of each bonded specimen from the bonding area, including the base metal and the reactive multilayers, were prepared. The calorimetry tests were carried out in accordance with the ASTM D 3418–15 standard under the same conditions as the tests on base metals.

Results and discussion

The microstructure of the base metal is depicted in Fig. 3. In Fig. 3a, the structure resulting from twinning is fully visible (indicated by the red circle), and as can be seen in Fig. 3b, the base metal has an equiaxed grain structure. Furthermore, the base metal contains titanium carbide deposits (Fig. 3c, d, and e), as indicated by points 1 and 2 in Fig. 3d, along with the corresponding EDS results detailed in Fig. 3e. Dispersed carbides within the Nitinol field are employed either to harden the matrix (Properties and Selection: Nonferrous Alloys and Special-Purpose Materials. 1990) or to change the martensitic transformation temperature (Nishida and Wayman 1987). In this way, the transformation temperatures can be controlled to be compatible with the human body temperature. Shogo et al. (Shugo et al. 1982) reported that as the carbon content in Ni–Ti-C alloy increases, the temperature of M_s decreases, and most of the carbon present in the alloy precipitates in the form of TiC in the NiTi-based ground phase.

The DSC test results for the base metal provide detailed phase transformation temperatures for Nitinol. The temperatures for Martensite start (M_s), Martensite finish (M_f), Austenite start (A_s), and Austenite finish (A_f) are determined to be 37.4 °C, 13 °C, 26.7 °C, and 30.1 °C, respectively. Additionally, mechanical parameters at the martensite phase have been documented in Table 1.

Figure 4 reveals the microstructure of the diffusion-bonded area of Nitinol without using the reactive multilayer at different magnifications. It is quite obvious that without the reactive multilayer, and with a low force (5 MPa) at the interface of the two parts at 900 °C, and a holding time of 60 min, no all-round bond was formed. However, Fig. 5a–d shows that a completely solid and smooth joint was formed between the multilayer and the Nitinol. This result is achieved by using reactive Ni/Ti multilayers in the diffusion bonding region as a supporting bonding element under the same bonding conditions. This effect also applies when bonding is carried out at lower temperatures, as shown in Fig. 5a–d. These images depict the shared interface of diffusion-bonded samples prepared at varying temperatures of 600 °C, 700 °C, and 800 °C. In all samples, a diffusion boundary is clearly visible (labeled as "diffusion interface" in Fig. 5a–c), which



Spectrum	C (wt. %)	Ti (wt. %)
1	10.48	89.52
2	9.95	90.05

Fig. 3 **a**, **b** Base metal optic microstructure in different magnifications, **c**, **d** Base metal SEM microstructure in different magnification, A: TiC deposit, **e** EDS analysis graph for the point of A

Table 1 Base metal mechanical properties

Material	Martensite phase Young's modulus (GPa)	Martensite phase Yield stress (MPa)	Martensite phase Ultimate tensile strength (MPa)	Total elongation (%)	Elastic elongation (%)	Plastic elongation (%)
NiTi	10	70	1346	33	29	4

widens with increasing temperature due to enhanced interdiffusion of Ni and Ti within the base metal.

In contrast, at lower temperatures (600, 700, and 800 °C), a dark layer forms in the joint region, which is an intermetallic compound other than NiTi and covers almost the entire joint area. According to the EDS analysis graphs in Fig. 6a–c and the results given in the tables, this dark intermetallic compound is identified as Ti_2Ni (Fig. 6a), which transforms into the TiNi phase (Fig. 6b and c) at higher temperatures (e.g., 900 °C). This transformation occurs due to the higher diffusion rate of nickel in TiNi compared to titanium in TiNi (Mokhles et al. 2024). Furthermore, by monitoring the thickness of the darker layer in Fig. 5a–d, it becomes apparent that as the temperature increases, the thickness of this layer decreases. In some areas of the sample joined at a temperature of 900 °C, this intermetallic compound begins to dissolve and is gradually replaced by TiNi (Figs. 5d and 6b and c).

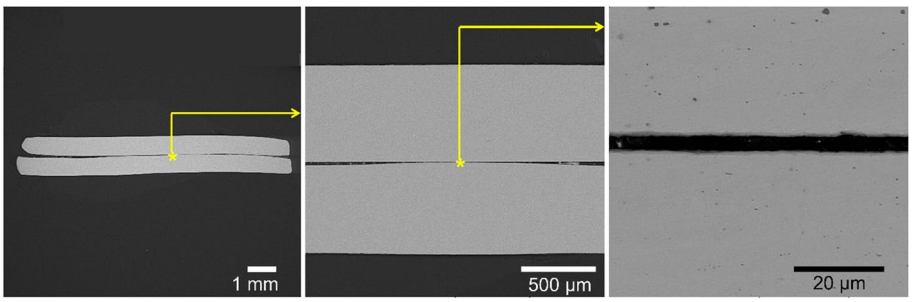


Fig. 4 Microstructure of diffusion bonded sample of Nitinol at 900 °C, 60 min, 5 MPa, and different magnification

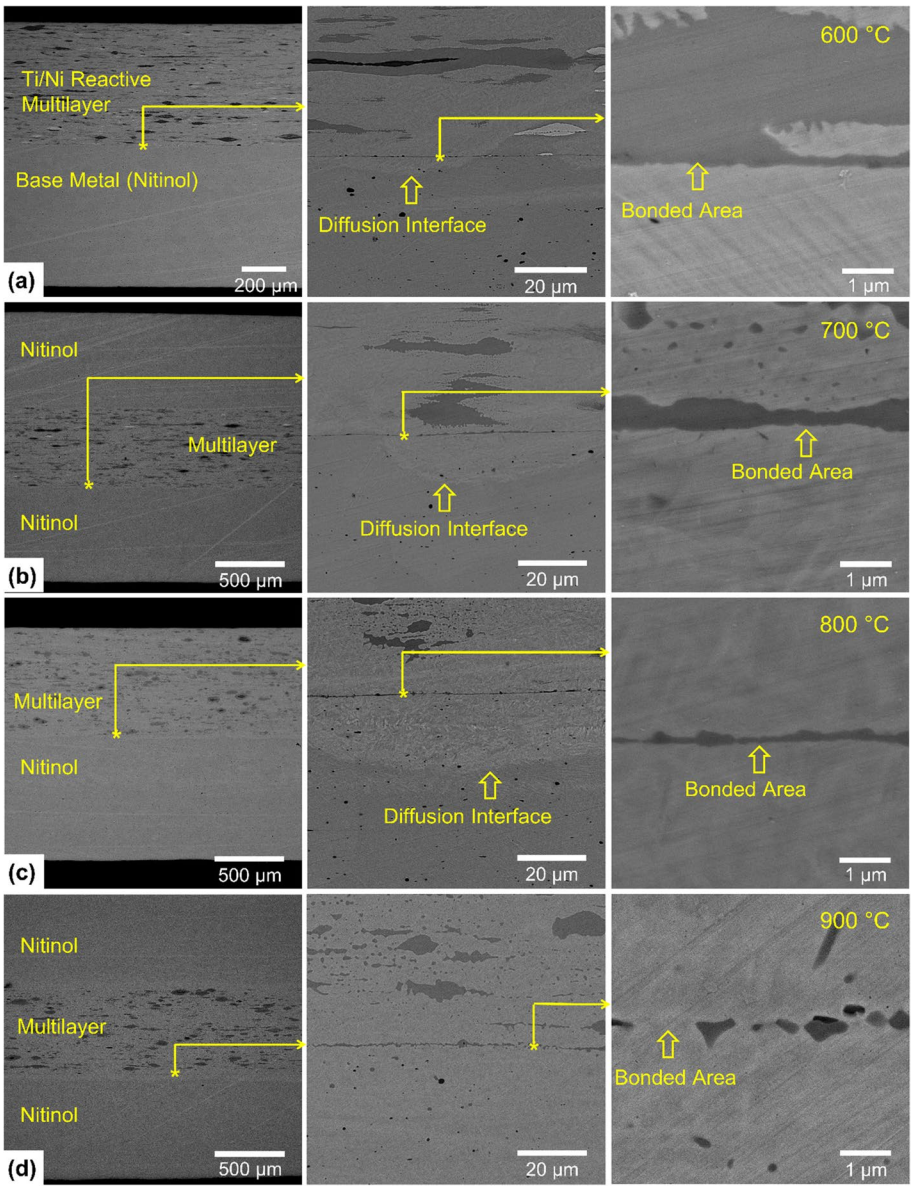
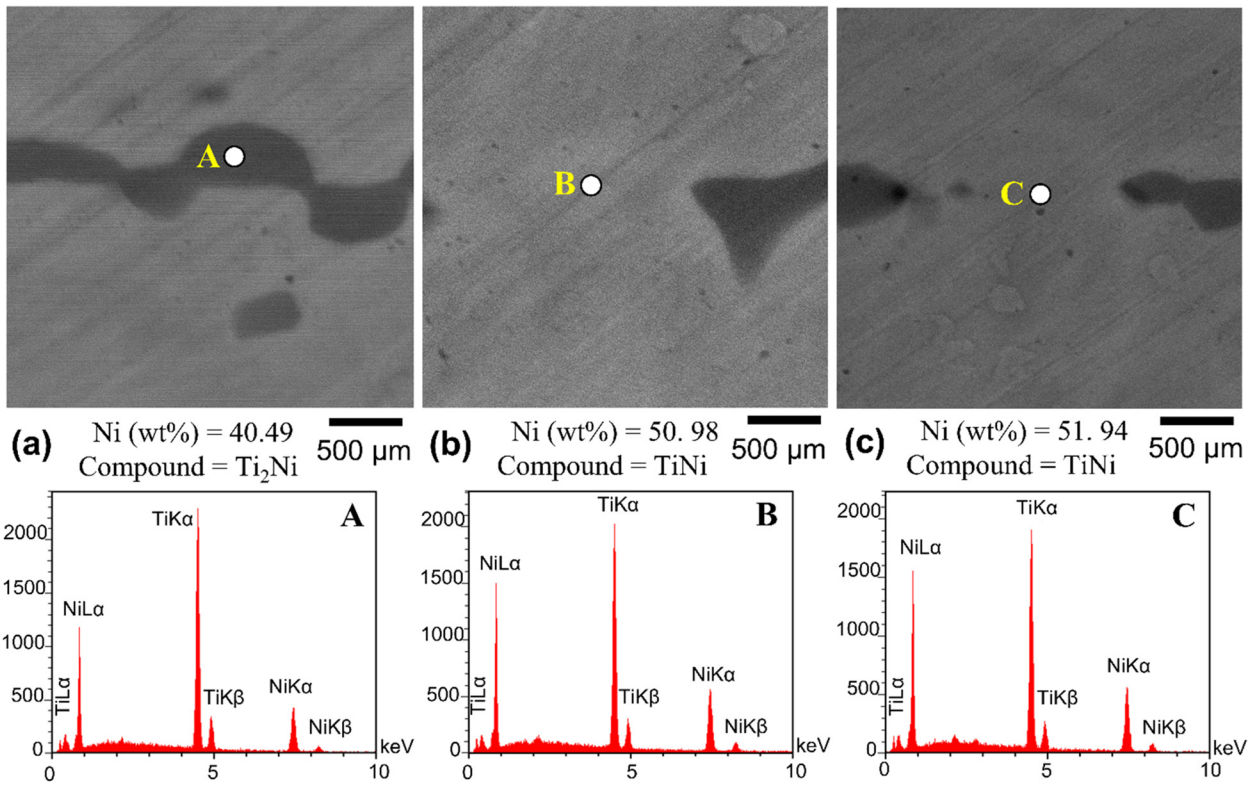


Fig. 5 Microstructure of diffusion bonded sample of Nitinol using Ti/Ni reactive multilayer at **a** 600 °C, **b** 700 °C, **c** 800 °C, **d** 900 °C, 60 min, 5 MPa, and different magnification



Spectrum A

Elt	Line	Int	Error	K	Kr	W%	A%	ZAF	Ox%	Pk/Bg	Class	LConf	HConf
Ti	Ka	947.5	1.0694	0.6012	0.5928	59.51	64.30	0.9962	0.00	49.36	A	58.80	60.21
Ni	Ka	207.3	0.5907	0.3988	0.3933	40.49	35.70	0.9714	0.00	19.81	A	39.46	41.52
				1.0000	0.9861	100.00	100.00		0.00				

Spectrum B

Elt	Line	Int	Error	K	Kr	W%	A%	ZAF	Ox%	Pk/Bg	Class	LConf	HConf
Ti	Ka	819.0	1.1478	0.4951	0.4882	49.02	54.10	0.9959	0.00	41.68	A	48.39	49.64
Ni	Ka	275.4	0.6070	0.5049	0.4978	50.98	45.90	0.9765	0.00	25.23	A	49.86	52.10
				1.0000	0.9860	100.00	100.00		0.00				

Spectrum C

Elt	Line	Int	Error	K	Kr	W%	A%	ZAF	Ox%	Pk/Bg	Class	LConf	HConf
Ti	Ka	794.9	0.9380	0.4853	0.4786	48.06	53.14	0.9959	0.00	40.06	A	47.43	48.68
Ni	Ka	278.0	0.7721	0.5147	0.5075	51.94	46.86	0.9770	0.00	25.51	A	50.81	53.08
				1.0000	0.9861	100.00	100.00		0.00				

Fig. 6 Microstructure of the joint interface in the diffusion bonded sample at 900 °C with the presence of Ti/Ni reactive multilayers for the EDS analysis of points **a** A, **b** B, and **c** C

The results of the shear strength tests (Fig. 7) show that the strength of the bonded joints increases with higher temperatures. This increase is attributed to the reduction in the thickness of the Ti_2Ni layer formed at the joint interface. Ti_2Ni represents a Ti–rich phase at the interface, and as the temperature increases, the tendency of Ti to diffuse from the Ti–rich phase into TiNi becomes dominant. Consequently, the Ti_2Ni thickness decreases under sufficient time and temperature conditions for diffusion bonding. Other studies have reported that a continuous layer of Ti_2Ni at the junction interface in nitinol is a major cause of brittle fracture (Akselsen 2010). The maximum strength observed is 35 MPa, which is achieved in the joint bonded at 900 °C. This is about 1.5 times the joint strength at the same temperature without the reactive multilayer. The optimum shear strength result is therefore achieved by producing a bonded joint at 900 °C with reactive Ni/Ti layers. This result is advantageous for both reasons: higher joint strength and formation of the desired phase structure, NiTi, at the joint interface.

Comparatively, in a study by Ma et al. (Ma, et al. 2016), which dealt with the diffusion bonding of Nitinol and Ti-6AL-4 V and used a Ti/Ni reactive multilayer with

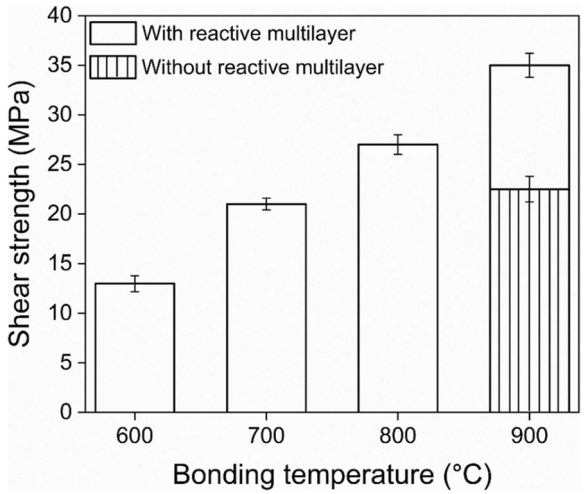


Fig. 7 Comparison diagram of the shear strength of the diffusion boned joint of Nitinol with and without the presence of Ti/Ni reactive multilayers

the same stoichiometry ratio of 1:1, a temperature of 800 °C and identical time and load, similar results were obtained, with shear strength around 25 MPa.

Nitinol, as a shape memory alloy, undergoes Martensitic to Austenitic transformation and vice versa. The transformation temperature of this unique alloy can be determined through a Differential Scanning Calorimetry (DSC) test. It is noteworthy that the presence of a peak in the calorimetric test indicates the retention of the shape memory effect in the diffusion-bonded joints facilitated by reactive Ti/Ni multilayers. The results of the calorimetry tests, along with the corresponding diagram during the heating stage (Fig. 8a) and the cooling stage (Fig. 8b), are presented. Table 2 contains the values of austenite to martensite and martensite to austenite transformation temperatures for the samples bonded using reactive multilayer at 900, 800, 700, and 600 °C, in comparison to the base metal of Nitinol before bonding, serving as a reference. The results demonstrate that diffusion bonding induces fundamental changes in the transformation temperature compared to the base metal in all investigated conditions. Notably, among the samples joined at different temperatures, the sample bonded at 800 °C exhibits the closest transformation temperature to that of the base metal.

While previous reports indicate an increase in the phase transformation temperature in the diffused area compared to the base metal during the fusion welding of NiTi (Falvo et al. 2005), our study focuses on assessing shape memory properties through calorimetric tests. Analysis of the obtained peak data reveals the persistence of these properties. Examination of transformation temperatures in the bonded samples under different conditions (Fig. 8a, b) indicates a decrease in the shape memory effect and alterations in transformation temperatures during sample heating across all bonded temperatures. Consequently, a training process becomes necessary for the connected parts to restore the shape memory property.

The shift in transformation temperature varies with aging times and temperatures, as illustrated in Nishida's diagram (Nishida and Wayman 1987). The aging process in Nitinol, at different temperatures, leads to the precipitation of various intermediate compounds. Therefore, the heating operation in diffusion bonding at different temperatures results in distinct transformation temperatures. To achieve a specific transformation temperature akin to the original base metal, it may be necessary to conduct homogenization followed by a specialized aging operation, as suggested by Nishida (Falvo et al. 2005). It is crucial to note that changes in shape memory properties due to heat, as caused by the welding process, are inherent in all fusion welding processes. However, the diffusion bonding process offers more control over these changes due to the absence of re-melting. In summary, the presence of shape memory properties in the joined area stands as one of the advantages of employing Ti/Ni reactive layers in the diffusion bonding of Nitinol. This property can be further enhanced through a training process for the joint part (Ikai et al. 1996).

Conclusion

The Ti/Ni reactive multilayer was produced by ARB processing for up to 10 cycles without any pre- or post-heat treatments. These reactive multilayers, formed during 10 ARB steps, were used as the optimal reactant multilayer to create diffusion-bonded joints at different bonding temperatures. In the second step, the prepared joints were studied for mechanical properties, joint microstructure, and joint shape memory effect, leading to the following results:

1 Investigating the structure of the joint area of Nitinol alloy with the presence of multilayer at different temperatures for 1 h and an applied stress of 5 MPa showed that, unlike the sample without reactive multilayer, the presence of multilayers causes complete bonding at the joined boundary of the two pieces. In these samples, the Ti_2Ni layer is formed at the border between the two parts, and its thickness decreases with the increase in the joining temperature. Therefore, in the sample joined at 900 °C, in some parts,

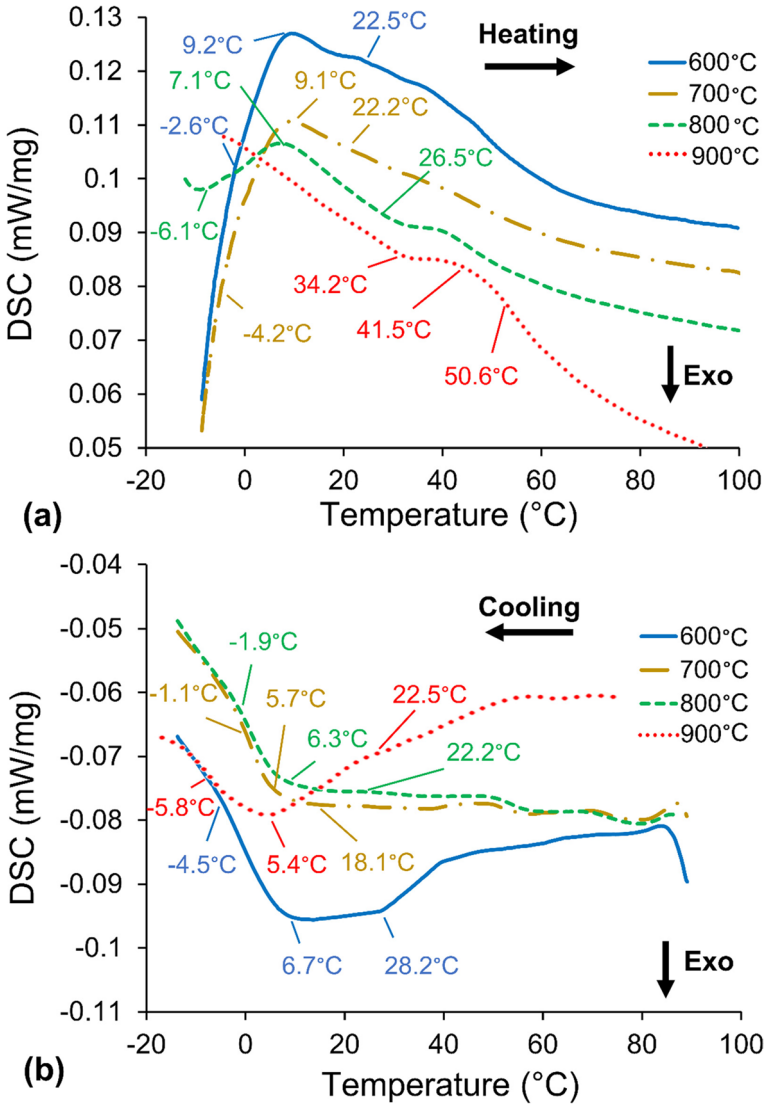


Fig. 8 DSC diagram of a) the heating (Martensite to Austenite transformation) and b) the cooling stage (Austenite to Martensite transformation) of the diffusion-bonded joints at 600, 700, 800, and 900 °C in the presence of Ti/Ni reactive multilayers

Table 2 The transformation temperatures of diffusion-bonded joints of Nitinol at 900, 800, 700, and 600 °C with the presence of Ti/Ni reactive multilayers compared to the base metal

Items	Description	A_s (°C)	A_f (°C)	M_s (°C)	M_f (°C)
1	Base metal—Nitinol	26.7	30.1	37.9	13
2	Diffusion bonded at 900 °C	34.2	50.6	22.5	−5.8
3	Diffusion bonded at 800 °C	−6.1	26.5	22.2	−1.9
4	Diffusion bonded at 700 °C	−4.2	22.2	18.1	−1.1
5	Diffusion bonded at 600 °C	−2.6	22.5	28.2	−4.5

this compound is completely removed and replaced by TiNi.

2 Examining the shear strength of the joint shows that the shear strength of the overlap joint increases with the temperature of the diffusion joining process. In the sample bonded at 900 °C with the presence of Ti/Ni reactive multilayers, the strength reaches 35 MPa, which is about 1.5 times compared to the sample without reactive multilayers under the same bonding conditions.

3 Examining the calorimetric test of the samples bonded with the help of reactive multilayers shows that the shape memory property is observed in the bonding area. The presence of shape memory

property in the bonded area is considered one of the main advantages of using Ti/Ni reactive multilayers in the diffusion bonding of Nitinol, a primary shape memory alloy used in industry.

4 Among the samples bonded at different temperatures, the sample connected at 800 °C shows a phase transformation temperature closer to the base metal.

Acknowledgements
The authors are grateful to Shiraz University, for supporting the study with basic infrastructure and facilities characterization involved in this research work.

Authors' contributions
MM: conceptualization, investigation, data curation, methodology, visualization, writing—original draft preparation; SMZ: conceptualization, resources, supervision, writing—review and editing; HD-M: conceptualization, resources, supervision, funding acquisition; TRD: supervision resources, writing—review and editing.

Funding
This work was supported by Shiraz University [grant number 95INT4M1974].

Availability of data and materials
The datasets generated and/or analyzed during the current study are available on request.

Declarations

Competing interests
On behalf of all authors, the corresponding author states that there is no conflict of interest.

Received: 18 April 2024 Accepted: 2 August 2024
Published online: 13 August 2024

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