www.czasopisma.pan.pl www.journals.pan.pl

Arch. Metall. Mater., Vol. 61 (2016), No 2B, p. 997–1001

DOI: 10.1515/amm-2016-0170

B. SZWED*#, M. KONIECZNY*

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF JOINTS OF TITANIUM WITH STAINLESS STEEL PERFORMED USING NICKEL FILLER

Diffusion brazing was performed between titanium (Grade 2) and stainless steel (X5CrNi18-10) using as a filler a nickel foil at the temperatures of 850, 900, 950 and 1000°C. The microstructure was investigated using light microscopy and scanning electron microscopy equipped with an energy dispersive X-ray system (EDS). The structure of the joints on the titanium side was composed of the eutectoid mixture $\alpha Ti+Ti_2Ni$ and layers of intermetallic phases Ti_2Ni, TiNi and $TiNi_3$. The stainless steel-nickel interface is free from any reaction layer at 850°C, above this temperature thin layer of reaction appears. The microhardness measured across the joints reaches higher values than for titanium and stainless steel, and it achieves value from 260 to 446 HV. The highest shear strength (214 MPa) was achieved for joints brazed at 900°C.

Keywords: diffusion brazing, titanium, stainless steel, microstructure, mechanical properties

1. Introduction

Joining of dissimilar materials, like titanium and stainless steel, that have different physical and chemical properties and producing joints characterised by good mechanical properties constitutes today a significant problem. Such types of joints have applications in aerospace, chemical and nuclear industries [1-4]. Conventional fusion welding is not a feasible technique to join these materials due to the different problems like formation of stress concentration at the bond interface and development of chemical heterogeneities [5-7]. Hence, diffusion brazing offers a feasible solution to overcome these problems [8]. However the direct bonding between titanium and stainless steel promotes residual stress at the interface region caused by mismatch of thermal expansions between those materials. Also, it leads to the formation of brittle intermetallic phases in the diffusion zone [9-11]. The use of appropriate intermediate materials can minimise the thermal expansion mismatch, reduce joining temperature and pressure, inhibit diffusion of undesired elements and reduce or eliminate the formation of brittle intermetallic phases [12]. Aluminium, copper, silver and their alloys were used previously as intermediate materials [13-16]. The literature survay shows that the copper layer of 0.1 mm thickness effectively blocks the diffusion of titanium to stainless steel only up to 900°C and if the bonding time is no longer than 30 minutes [17,18]. Previous attempts [19] and literature data [20,21] report that pure nickel and nickel alloys also may be considered as a useful filler material between titanium and stainless steel due to the satisfactory corrosion resistance at high temperature applications comparing to copper that also is often used as an intermediate material. Nickel has substantial solid solubility in iron. Kamat et al. have reported that nickel-stainless steel (Ni-SS) diffusion couple is free form intermetallic compounds [22]. However, Kundu and Chatterjee have shown that the Ni-SS diffusion interface is free from intermetallic compounds up to 850°C [23,24].

The present study demonstrates the feasibility of diffusion brazing of commercial pure titanium and stainless steel using nickel as an intermediate material at varying brazing temperatures, with focus on the microstructure and mechanical properties of the diffusion brazed joints.

2. Experimental procedure

The base materials used in this work were pure titanium (Grade 2) and stainless steel (X5CrNi18-10), both received in the form of cylindrical rods having 8 mm diameter and 2000 mm length, and nickel foil of 0.1 mm thickness. Cylindrical specimens of 8 mm diameter and 10 mm length were machined from the titanium and stainless steel rods. The circular profiles discs with 8 mm diameter were excised from the nickel foil. The nominal chemical composition and room temperature mechanical properties of these materials are given in Table 1.

---

* KIELCE UNIVERSITY OF TECHNOLOGY, FACULTY OF MECHATRONICS AND MECHANICAL ENGINEERING, DEPARTMENT OF APPLIED COMPUTER SCIENCE AND ARMAMENT ENGINEERING, KIELCE, POLAND

Corresponding author: bartlomiej_szwed@o2.pl

TABLE 1

Chemical compositions and mechanical properties of the base materials

Material	**Chemical composition (wt. %)**	**Mechanical properties**		
		YS (MPa)	**UTS (MPa)**	**A (%)**
Titanium Grade 2	Ti: 99,654; Fe: 0,171; C: 0,024; N: 0,008; O: 0,142; H: 0,001	350	420	38
Stainless steel X5CrNi18-10	Fe: 77,99; C: 0,025; Mn: 1,460; Si: 0,390; P: 0,038; S: 0,012; Cr: 18,150 ; Ni: 8,050; Mo: 0,380	480	620	26
Nickel Ni 99,6	Ni: 99,57; Cu: 0,11; Co: 0,09; Si: 0,08; Mg: 0,07; Fe: 0,07; Al: 0,01	146	448	43

The faces of the cylinders were prepared by conventional grinding and polishing techniques and final polishing was made with 0.5 µm alumina suspension. To remove oxide layers from the base material, the samples were etched in acid solutions: titanium in an aqueous 5% solution of HF, stainless steel in an aqueous 10% solution of HCl, nickel in an aqueous 10% solution of HNO_3. The mating surfaces of the samples were kept in contact with steel clamp (Fig. 1) and inserted in a vacuum chamber.

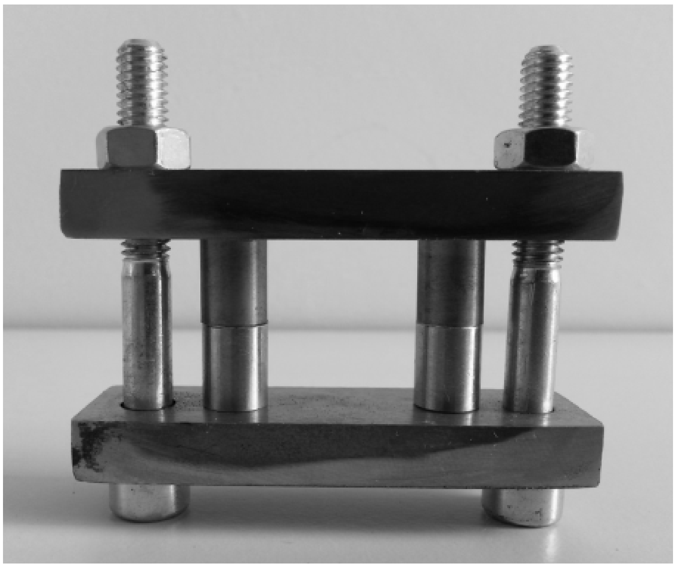

Fig. 1. Samples placed in a steel clamp

The compressive stress of 2 MPa along the longitudinal direction was applied at room temperature. Diffusion brazing was carried out in a vacuum furnace Czylok PRC 77/1150 (Fig. 2) in the temperature range from 850 to 1000°C for 60 minutes with a vacuum of 10^{-3} Pa. The samples were cooled with the furnace. The specimens for metallographic examination were cut out longitudinally and their surfaces were prepared by conventional techniques, using sandpapers of 180 to 1200 grit and alumina suspension with a grain size of 0.5 µm. The titanium side was etched in an aqueous solution of 95 ml H_2O and 5 ml HF. The stainless steel substrate was etched with a mixture of 90 ml C_2H_5OH, 10 ml HCl and 3 g $FeCl_3$. A solution consisting of 50 ml $C_2H_4O_2$ and 50 ml HNO_3 was used for etching the nickel interlayer.

Fig. 2. Czylok PRC 77/1150 Vacuum furnace

The samples were observed in a light microscope Nikon Eclipse MA200 to reveal the structural changes due to diffusion. The polished surfaces of the brazed couples were also examined in a scanning electron microscope (SEM) JEOL JMS-5400 to obtain finer structural details in the diffusion zone. The composition of the raction layers was determined in atomic percent using Oxford Instruments ISIS energy dispersive X-ray spectrometer (EDS) attached to the SEM. The results of the EDS analysis were compared with the binary phase diagrams of basic components. The Matsuzawa MMT microhardness tester was used to examine the hardness along the cross-section of the joints under load of 0.196 N for a dwelling time of 10 seconds. The shear strength of the brazed joints was evaluated at room temperature using an LabTest 5.20SP1 testing machine at a crosshead speed of 10 mm/min. Five samples were tested for each processing parameter.

3. Results and discussion

The microstructures of the diffusion brazed joints revealed by the light microscope are shown in Fig. 3.

The results of the metallographical examination show that the diffusion interfaces are free from cracks and interface lines are clearly visible. However, Kirkendall voids were observed at the borders of materials. The width of the diffusion zone on the boundaries along the joined materials increased with the brazing temperature until the liquid phase appearance. The titanium side of the joint is characterized by the α-β Ti structure. Because nickel is a β stabilizing element it lowers the eutectoid transformation temperature of Ti [25]. The occurrence of acicular α-β Ti results from the decomposition of β Ti during cooling with the vacuum furnace. Three distinct reaction layers were observed

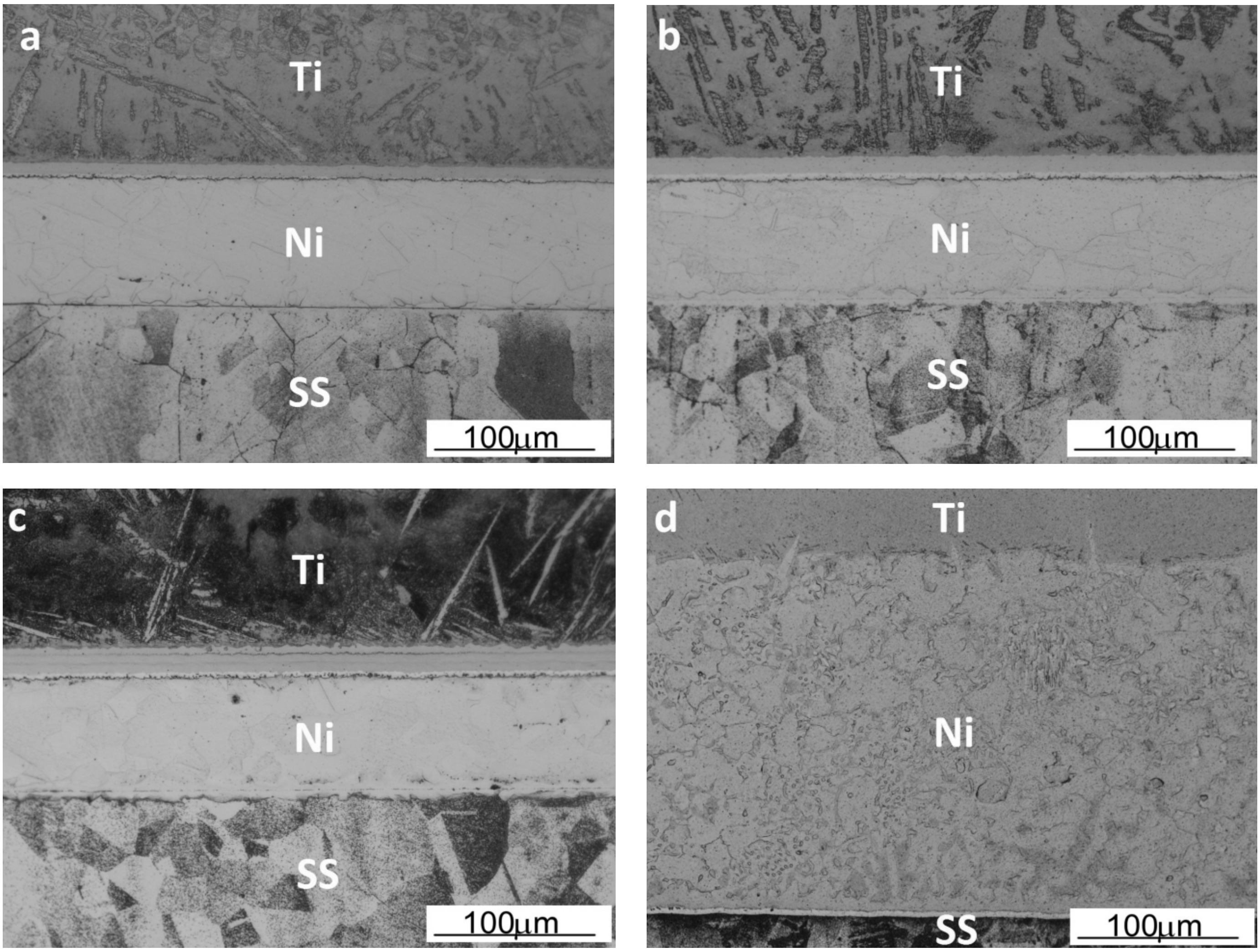

Fig. 3. Light micrograph of the joints prepared at a) 850, b) 900, c) 950 d) 1000°C for 60 minutes

at the Ti-Ni interface. The thickness of the reaction products on the Ti-Ni side increases with rising of the brazing temperature. The diffusion zone at the stainless steel-nickel interface is thinner than that at Ti-Ni side. The diffusion zone at the SS-Ni interface at brazing temperature 850°C was planar and very thin. The presence of distinct layer was revealed when the brazing temperature increased to 900°C. The SEM investigation of the joints was performed in order to reveal more details in the reaction layers of the joint. Scanning electron microscopic images of the diffusion brazed joints are shown in Fig. 4

At the titanium-nickel interface three distinct reaction layers were observed for 850-950°C processing temperature. The first reaction layer adjacent to the titanium side consisted of Ti (69.4-70.9 at.%) and Ni (bal.). According to the Ti-Ni binary phase diagram it is likely a Ti_2Ni intermetallic compound. The brightest layer on the nickel side consisted of Ni (73.1-75.9 at.%) and Ti (bal.). This composition corresponds to the $TiNi_3$ intermetallic phase. Between the two intermetallic phases, another reaction layer, consisted of Ti (50.5-51.3 at.%) and Ni (bal.), was revealed. This is likely the TiNi phase. At the 1000°C brazing temperature, a significant change was observed. The well-defined islands (shown in Fig. 4d) were composed of Ti (93.4 at.%) and Ni (5.6 at.%.) with small additions of Fe (1 at.%.). This microstructure is likely a mixture of αTi+Ti_2Ni. The bright area corresponds to the Ti_2Ni intermetallic phase with small amount of Fe (1.2 at.%.). On the stainless steel nickel side, for the 850°C brazing temperature, no reaction products were observed in the diffusion zone. The presence of Fe (10 at.%.), Cr (0.4 at.%.) and Mn (0.6 at.%.) in nickel indicates the substantial interdiffusion of those elements. The presence of a solid solution γFe+Ni between nickel and stainless steel was observed at the 900°C processing temperature, until the appearance of the liquid phase. At the stainless steel interface four distinct reaction layers were observed for the 1000°C brazing temperature (Fig. 2d). According to the literature the first layer adjacent to the steel was identified as a mixture of $\lambda + \chi + \alpha$Fe phases [20,21]. As reported by Kundu and Chatterjee [23,24] the nickel concentration in this area is too low to form any nickel base intermetallic compound. The brightest layer has been recognized as $\lambda + \alpha$Fe phase combination. The fourth was identified as a combination of FeTi + Ti.

Microhardness measurements of titanium substrate, interface zone and steel substrate were performed for all processed samples. The hardness values for the base materials were as follows: for titanium 260 HV, for nickel 124 HV and for stainless

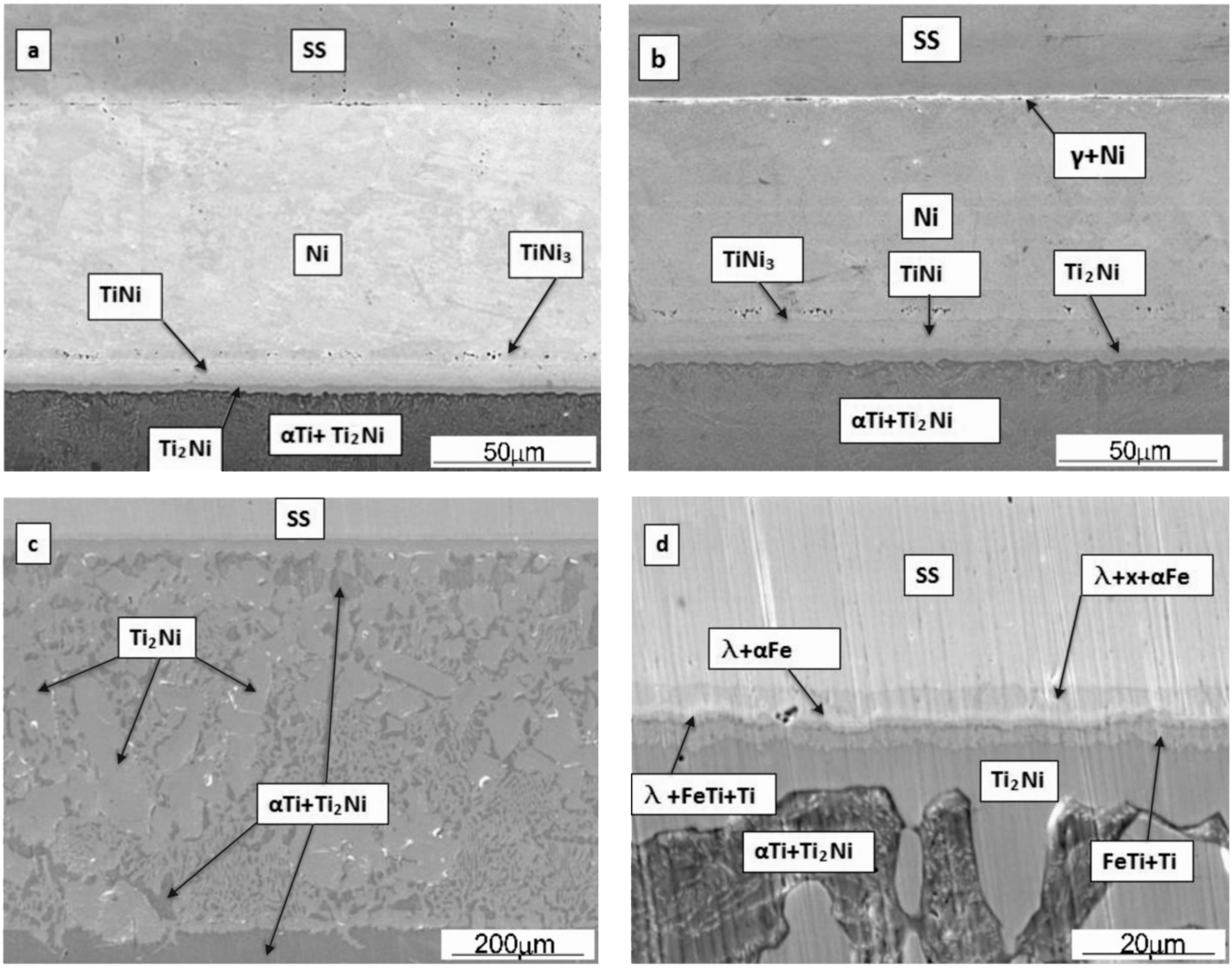

Fig. 4. SEM images of the brazed joints processed at a) 850, b) 900, c) and d) 1000°C for 60 minutes

steel 202 HV. The maximum hardness values in the range of 260 to 446 HV were recorded at the titanium-nickel interface due to the formation of the Ti_2Ni, TiNi and $TiNi_3$ intermetallic phases. The hardness values for those phases were as follow: 446 HV for Ti_2Ni, 322 HV for TiNi and 345 HV for $TiNi_3$. It was observed that the hardness of the Ni-SS interface, for bonding temperatures from 850 to 950°C, reached a maximum value of 132 HV. Such a value resulted from the presence of a solid solution γFe + Ni. However, when the brazing temperature approached 1000°C the increase in the hardness to the 420 HV was observed. It was likely caused by the occurrence of the FeTi intermetallic phase.

The shear strength of the diffusion brazed joints with the change in bonding temperature is given in Fig. 5.

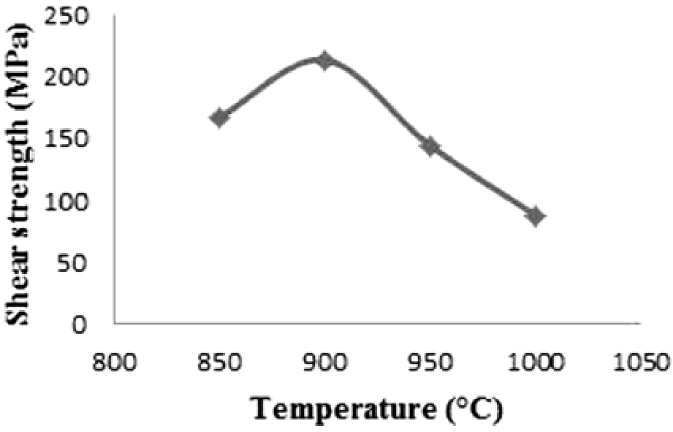

Fig. 5. Shear strength for diffusion brazed joints processed for 60 min

At the lowest processing temperature of 850°C, the shear strength reached a value of 167 MPa. This indicates that the process proceeded correctly and the diffusion occurred between nickel filler metal and the two substrates. With an increase in the joining temperature to 900°C, the shear strength increased to its maximum value of 214 MPa. At this processing temperature, the atomic diffusivity of the chemical species is enhanced, which leads to the increase in the shear strength. With an increase in bonding temperature to the 950°C can be observed an increase in the thickness of the intermetallic phases at the Ti-Ni interface. The growth of the those intermetallics reduce the bond strength to

144 MPa. At 1000°C brazing temperature the Fe-Ti brittle intermetallics formed at the Ni-SS interface. Because of the appearance of a liquid phase the nickel interlayer could not block the diffusion of titanium to the stainless steel side. Thus, the bond strength decreased and attained its lowest value of 88 MPa.

4. Conclusions

The investigations of the diffusion brazed joints revealed the following:

1. Diffusion brazing temperature is a critical factor controlling the microstructure. The diffusion zone on the titanium side of the joint becomes wider with the increase in temperature while on the stainless steel side it is planar and very thin until the liquid phase appears.
2. The intermetallic layers Ti_2Ni, TiNi, $TiNi_3$ were observed at the titanium nickel side of the diffusion joint, however the TiNi, and $TiNi_3$ phases were not found at 1000°C. There where only islands of $\alpha Ti + Ti_2Ni$ in the Ti_2Ni matrix. The thicknesses of the Ti_2Ni, TiNi and $TiNi_3$ intermetallic layers increases with the increase in the brazing temperature.
3. The stainless steel-nickel interface is free from any reaction layer at 850°C. Above this temperature a thin layer of a solid solution $\gamma Fe + Ni$ appears between nickel and stainless steel. The mixture of $\lambda + FeTi + Ti$, $FeTi + Ti$, $\lambda + \alpha Fe$ and $\lambda + \chi + \alpha Fe$ phases was formed at 1000°C.
4. The nickel interlayer of 0.1 mm thickness blocked the diffusion of titanium to stainless steel side up to 950°C. After the appearance of a liquid phase titanium atoms can diffuse to the stainless steel and form the FeTi intermetallic phase.
5. The microhardness test across the joints indicates that the hardness in the interfaces reaches higher values than for titanium and stainless steel. The hardness values were in the range 260 to 446 HV.
6. The maximum shear strength of 213.7 MPa was obtained for the diffusion brazed joints performed at 900°C; the bonding strength decreased with the rising of the joining temperature due to the increase in width of intermetallics phases at the Ti-Ni side and the formation at the 1000°C FeTi phase at the SS-Ni interface. The lowest shear strength of 87.9 MPa was obtained for samples brazed at the highest temperature.

REFERENCES

[1] American Welding Society Staff, Brazing Handbook ed **IV**, Miami, (1991).
[2] M. Ghosh, S. Chatterjee, Mat. Sci. Eng. A-struct. **358**, 152 (2003).
[3] A. Winiowski, Arch. Metall. Mater. **52**, 593 (2007).
[4] A. Elrefaey, W. Tillmann, J. Mater. Sci. **42**, 9553 (2007).
[5] S. Kundu, S. Chatterjee, D. Olson, B. Mishra, Metall Mater Trans A. **39**, 2106 (2008).
[6] A. Winiowski, Arch Metall Mater. **55**, 991 (2010).
[7] A. Elrefaey, W. Tillmann, J. Mater. Process. Tech. **209**, 4842 (2009).
[8] A. Dziadoń, R. Mola, L. Błaż, Arch. Metall. Mater. **56**, 677 (2011).
[9] H. Kato , S. Abe, T. Tomizawa, J. Mater. Sci. **32**, 5225 (1997).
[10] B. Aleman, I. Gutierrez, J.J. Urcola, Mater. Sci. Tech. Ser. **9**, 633 (1993).
[11] M. Ferrante, E.V. Pigoretti, J. Mater. Sci. **37**, 2825 (2002).
[12] R.K. Shiue, S.K. Wu, C.H. Chan, C.S. Huang, Metall. Mater. Trans. A. **37**, 2207 (2006).
[13] M. Konieczny, B. Szwed, R. Mola, METAL 2015 conference, **151** (2015).
[14] B. Szwed, M. Konieczny, J. Mater. Manuf.. Eng. **67**, 21 (2014).
[15] E. Atasoya, N. Kahramanb, Mater. Charact. **59**, 1481 (2008).
[16] A. Winiowski, Kovove Mater. **51**, 19 (2013).
[17] M. Konieczny, R.Mola, Steel Res Int. **79**, 499 (2008).
[18] M. Konieczny, Kovove Mater. **48**, 47 (2010).
[19] B. Szwed, M. Konieczny, R. Mola, METAL 2015 Conference, **160** (2015).
[20] S. Sam, S. Kundu, S. Chatterjee, Mater Design. **40**, 237 (2012).
[21] S. Kundu, S. Chatterjee, Mater. Sci. Eng. **425** 107 (2006).
[22] G.R. Kamat, Weld. J. **67**, 44 (1988).
[23] S. Kundu, S. Chatterjee, J. Mater. Sci. **42**, 7906 (2007).
[24] S. Kundu, S. Chatterjee, Mater. Charact. **59**, 631 (2008).
[25] I.J. Polmear, Light Alloys ed **IV**, Melbourne, (2005).