

## CHARACTERIZATION OF TRANSIENT LIQUID-PHASE BONDING BETWEEN TITANIUM AND STAINLESS STEEL USING NICKEL INTERLAYER

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### Abstract

In the present investigation, transient liquid-phase bonding was performed between pure titanium (Grade 2) and stainless steel (X5CrNi 18-10) using 0.1 mm thick nickel foil as an intermediate material. The process was carried out at the temperatures of: 850, 875, 900, 925, 950 and 1000 °C for 60 minutes under the compressive stress of 2 MPa in a vacuum. The effects of temperature on the microstructure diffusion brazed joints were characterized in the optical and scanning electron microscopes. The inter diffusion of chemical species across the diffusion interface were evaluated by electron probe microanalysis. The structural examinations have shown that temperature is critical factor to control the microstructure. The diffusion zone on the borders of the joined materials becomes wider with the increase in temperature. The structure of the joints from the titanium site was composed of the eutectoid mixture  $\alpha$ Ti+Ti<sub>2</sub>Ni and layers of intermetallic phases Ti<sub>2</sub>Ni, TiNi and TiNi<sub>3</sub>. The stainless steel-nickel interface is free from any reaction layer up to 875 °C, above this temperature thin layer of reaction appears. The microhardness test across the joints indicates that the hardness in the interfaces reaches higher value than for titanium and stainless steel, and it achieves value from 320 to 528 HV.

**Keywords:** transient liquid-phase bonding, diffusion brazing, titanium, stainless steel, nickel

### 1. INTRODUCTION

In the recent years, there is a strong interest to produce joints between different dissimilar materials to satisfy require of modern industries. Titanium and stainless steel have satisfactory specific strength, high melting point and excellent corrosion resistance. Because of that aerospace, chemical and nuclear industries strongly demand joints between titanium and stainless steel [1-3]. Many titanium joining methods, including the commonly employed joining processes, welding, brazing and soldering, all face the reactive nature of titanium. Joining by the conventional techniques also results in segregation of chemical species, stress concentration sites and formation of intermetallics at interfaces due to very low solubility of Ti and Fe [ 1, 4]. Diffusion brazing is one of the viable solutions that allows to combine dissimilar materials without gross microscopic distortion and with minimum dimensional tolerance [1, 5]. The literature reports that joints produced by direct bonding between titanium and stainless steel (Ti-SS) showed the formation of Fe, Cr, Ni and Ti base intermetallic products [6, 7]. The formation of an intermetallic phase also causes the problem of residual stresses resulting from thermal expansion mismatch [8]. The use of appropriate intermediate materials can minimise thermal expansion mismatch, reduce joining temperature and pressure, inhibit diffusion of undesired elements and reduce or avoid the formation of brittle intermetallic phases [9]. Reports in the literature show that the copper layer of 0.1 mm thickness effectively blocks the diffusion of titanium to stainless steel up to 900 °C if the bonding time is no longer than 30 minutes [10, 11]. In previous attempt of diffusion bonding was shown that copper does not form any intermetallic phase with iron, but due to the fact that the process time was 60 minutes it was observed the formation of the Fe-Ti phases [12, 13]. Literature reports that pure nickel and nickel alloys can be considered as a useful filler material between titanium and stainless steel due to satisfactory corrosion resistance for application at high temperature as compared to copper as an intermediate material [14-16]. Nickel has substantial solid solubility in iron and Kamat et al.

Have reported that nickel-stainless steel diffusion couple is free from intermetallic compounds [17]. However, Kundu and Chatterjee have shown that the Ni-SS diffusion interface is free from intermetallic compounds up to 850 °C [18, 19]. The present investigation reports transient liquid-phase bonding of titanium and stainless steel using nickel as an interlayer, with a special focus on the interface microstructure of the diffusion brazed joints.

## 2. EXPERIMENTAL PROCEDURE

### 2.1. Specimen preparation

In the present investigation pure titanium (Grade 2) and stainless steel (X5CrNi 18-10) were used in the form of cylindrical rods having 8 mm diameter and 2000 mm length and nickel foil of 0.1 mm thickness. From the titanium and stainless steel rods there were machined cylindrical specimens of 8 mm diameter and 10 mm length. The nominal chemical composition and room temperature mechanical properties of these materials are given in Table 1.

**Table 1** Chemical compositions and mechanical properties of the base materials (accordingly to certificates)

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 (X5CrNi 18-10)	Fe: 71,495; 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	945	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. There were cut circular profiles from the nickel foil having 8 mm diameter. 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<sub>3</sub>. The mating surfaces of the samples were kept in contact with steel clamp and inserted in a vacuum chamber. The compressive stress of 2 MPa along the longitudinal direction was applied at room temperature. Diffusion brazing was carried out in vacuum furnace Czylok PRC 77/1150 in the temperature range from 850 to 1000 °C for 60 minutes with a vacuum of 10<sup>-3</sup> Pa. The samples were cooled with the furnace.

### 2.2. Microstructural characterization and microhardness measurements

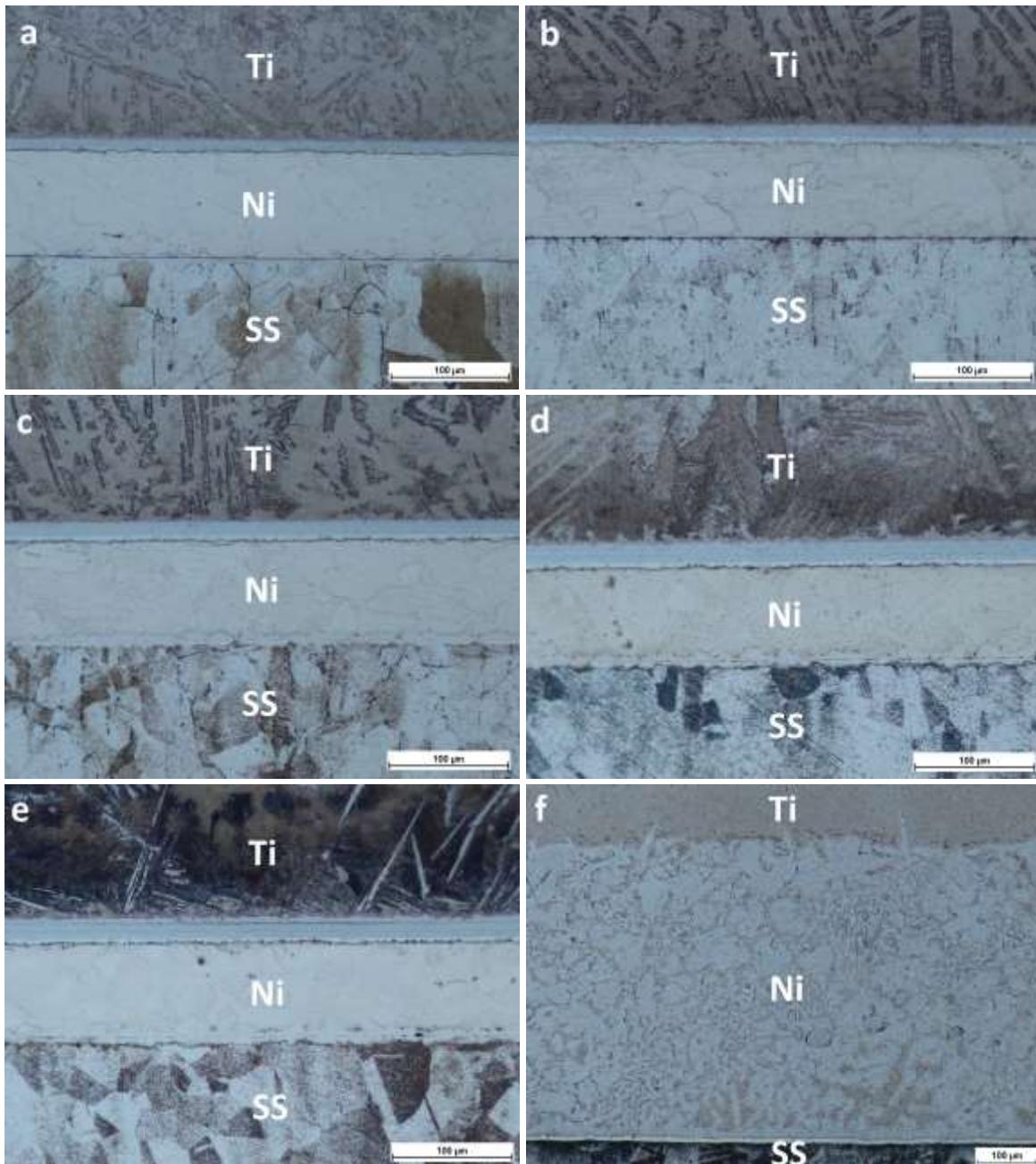
In order to examine, the specimens were cut longitudinally and the surfaces were prepared using conventional metallographic techniques. The titanium side was etched in an aqueous solution of 95 ml H<sub>2</sub>O and 5 ml HF. The stainless steel substrate was etched with a mixture of 90 ml C<sub>2</sub>H<sub>5</sub>OH, 10 ml HCl and 3 g FeCl<sub>3</sub>. A solution consisting of 50 ml C<sub>2</sub>H<sub>4</sub>O<sub>2</sub> and 50 ml HNO<sub>3</sub> was used for etching nickel interlayer. The samples were observed in optical microscope Nikon Eclipse MA200 to reveal the structural changes due to diffusion. The polished surfaces of the brazed couples were examined in a scanning electron microscope (SEM) JEOL JMS-5400 to obtain finer structural details in the diffusion zone. The composition of the reaction layers was determined in atomic percent using Oxford Instruments ISIS energy dispersive X-ray

spectrometer (EDS). The results of the microprobe 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.

### 3. RESULTS AND DISCUSSION

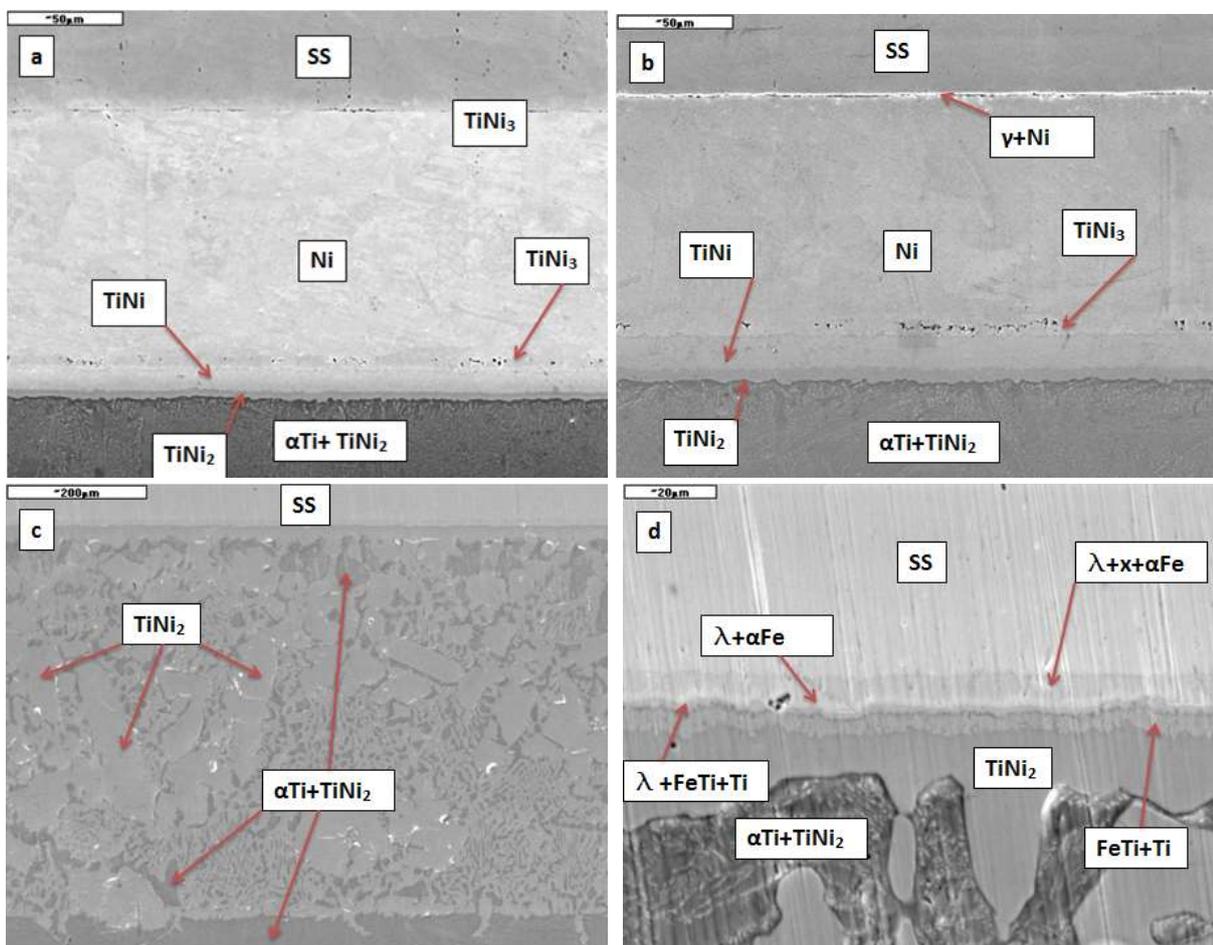
#### 3.1. Influence of diffusion brazing temperature on the microstructure of the joint

Brazed joints were successfully formed for all temperatures. The optical micrographs of the brazed assemblies are shown in Fig.1.



**Fig. 1** Optical micrograph of the joints prepared at a) 850, b) 875, c) 900, d) 925, e) 950 f) 1000 °C for 60 minutes.

From the micrographs, it can be seen that the diffusion interfaces are free from cracks and interface lines are clearly visible. However, at the borders of materials were observed Kirkendall voids. The wide of the diffusion zone on the boundaries with joined materials grown with an increase in bonding temperature. The titanium-nickel site is characterized by the  $\alpha$ - $\beta$  Ti structure. Because nickel is a  $\beta$  stabilizing element it lowers the eutectoid transformation temperature of Ti [20] and the acicular  $\alpha$ - $\beta$  Ti occurs from the decomposition of  $\beta$  Ti during cooling with the vacuum furnace. Three distinct reaction layers have been observed at the Ni-Ti interface. The thickness of the reaction products at the Ti-Ni side increases with increase in the brazing temperature. The diffusion zone at the stainless steel-nickel interface is smaller than that at Ti-Ni side. At the SS-Ni interface at brazing temperature from 850 to 875 °C planar and very thin diffusion layer was received. The presence of significant layer was revealed when the brazing temperature exceeded 900 °C. In order to further characterize the reaction layers of the joint, a SEM image of the transition joints was performed on the reaction layers. Scanning electron microscopic images of the diffusion brazed joints are shown in Fig. 2.



**Fig. 2** SEM images of transition joints processed at a) 850, b) 925, c) and d) 1000 °C for 60 minutes.

At the titanium-nickel interface three distinct reaction layers have been observed for 850-950°C processing temperature. The first reaction layer neighboring to titanium side consists of Ti (69.4-70.9 at.%) and Ni ( bal.). According to the Ti-Ni binary phase diagram it is  $Ti_2Ni$  intermetallic compound. The brightest layer at the nickel side consists of Ni (73.1-75.9 at.%) and Ti ( bal.). The composition indicates the presence of  $TiNi_3$  intermetallic phase. In between those two intermetallic compounds, it is present another reaction layer consists of Ti (50.5-51.3 at.%) and Ni ( bal.), this is the  $TiNi$  phase. Titanium form a combinations of bright and dark areas having the combination of of Ti (89-92.4 at.%) and Ni ( bal.), while nickel is enriched with a

small quantity of titanium (0.5 at.%). At 1000 °C brazing temperature, a significant change has been observed. The deeply shaded islands (shown in Fig. 2d) are composed of Ti (93.4 at.%) and Ni (5.6 at.%) with additions of Fe (1 at.%) which is a mixture of  $\alpha\text{Ti}+\text{Ti}_2\text{Ni}$ . The bright area is  $\text{Ti}_2\text{Ni}$  intermetallic phase with small amount of Fe (1.2 at.%). At the stainless steel nickel side up to the 900 °C brazing temperature, reaction products were not 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. Above 875 °C processing temperature can be observed a presence of a solid solution  $\gamma\text{Fe}+\text{Ni}$  between nickel and stainless steel, until the appearance of the liquid phase. At the stainless steel interface four distinct reaction layers have been observed for 1000 °C brazing temperature (Fig. 2d). According to the literature studies the first layer neighboring to the steel was identified as a phase mixture of  $\lambda+\chi+\alpha\text{Fe}$ . In accordance with Kundu and Chatterjee [18,19] in this area nickel concentration is too low to form any nickel base intermetallic compound. The brightest layer has been recognized as  $\lambda+\alpha\text{Fe}$  phase combination. The next to the  $\lambda+\alpha\text{Fe}$ , a light shaded reaction layer which is a phase mixture of  $\lambda+\text{FeTi}+\text{Ti}$ . The fourth was identified as a combination of  $\text{FeTi}+\text{Ti}$ .

### 3.2. Influence of diffusion brazing temperature on the hardness of the joints

Microhardness analyses of the brazed joints showed that the maximum hardness in the range of 320 to 528 HV were achieved at the titanium-nickel interface due to the presence of the  $\text{Ti}_2\text{Ni}$ ,  $\text{TiNi}$  and  $\text{TiNi}_3$  intermetallic phases. At the stainless steel-nickel hardness was in the range of 170 to 177 HV. In the middle of the joints hardness value were in the range of 89 to 94 HV. Hardness values increased with the increase in brazing temperature.

## CONCLUSIONS

The transient liquid-phase bonding of titanium Grade 2 to stainless steel X5CrNi 18-10 with 0.1 mm nickel interlayer has been performed in the temperature range of 850 to 1000 °C for 60 minutes under 2 MPa uniaxial load in vacuum furnace. The characterization of the diffusion brazed joints reveals the following:

1. Diffusion brazing temperature is critical factor to control the microstructure. The diffusion zone in the titanium site of the joint becomes wider with the increase in temperature while in the stainless steel side it is planar and very thin.
2. The intermetallic layers  $\text{Ti}_2\text{Ni}$ ,  $\text{TiNi}$ ,  $\text{TiNi}_3$ , were observed at the titanium nickel side of the diffusion joint, however the presence of  $\text{TiNi}$ ,  $\text{TiNi}_3$  were not found at 1000 °C. There where only islands of  $\alpha\text{Ti}+\text{Ti}_2\text{Ni}$  in a matrix of  $\text{Ti}_2\text{Ni}$ . The thicknesses of the  $\text{Ti}_2\text{Ni}$ ,  $\text{TiNi}$  and  $\text{TiNi}_3$  intermetallic layers increases with increase in the brazing temperature.
3. The stainless steel nickel interface is free from any reaction layer up to 875 °C. Above this temperature appears thin layer of a solid solution  $\gamma\text{Fe}+\text{Ni}$  between nickel and stainless steel. At 1000 °C there were formed a phase mixtures  $\lambda+\text{FeTi}+\text{Ti}$ ,  $\text{FeTi}+\text{Ti}$ ,  $\lambda+\alpha\text{Fe}$ ,  $\lambda+\chi+\alpha\text{Fe}$ .
4. The nickel interlayer of 0.1 mm thickness can completely block the diffusion of titanium to stainless steel side up to 950 °C. During the appearance of a liquid phase titanium atoms can cross to the stainless steel and form the  $\text{FeTi}$  intermetallic phase.
5. The microhardness test across the joints indicates that the hardness in the interfaces reaches higher value than for titanium and stainless steel. It achieves value from 320 to 528 HV.

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