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+$\text{Ti}_2\text{Ni}$ and layers of intermetallic phases $\text{Ti}_2\text{Ni}$, $\text{TiNi}$ and $\text{TiNi}_3$. 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
### TABLE 1

<table>
<thead>
<tr>
<th>Material</th>
<th>Chemical composition (wt. %)</th>
<th>Mechanical properties</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>YS (MPa)</td>
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<tr>
<td>Titanium Grade 2</td>
<td>Ti: 99.654; Fe: 0.171; C: 0.024; N: 0.008; O: 0.142; H: 0.001</td>
<td>350</td>
</tr>
<tr>
<td>Stainless steel</td>
<td>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</td>
<td>480</td>
</tr>
<tr>
<td>Nickel Ni 99.6</td>
<td>Ni: 99.57; Cu: 0.11; Co: 0.09; Si: 0.08; Mg: 0.07; Fe: 0.07; Al: 0.01</td>
<td>146</td>
</tr>
</tbody>
</table>

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 HNO₃. The mating surfaces of the samples were kept in contact with steel clamp (Fig. 1) 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 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 H₂O and 5 ml HF. The stainless steel substrate was etched with a mixture of 90 ml C₂H₂OH, 10 ml HCl and 3 g FeCl₃. A solution consisting of 50 ml C₂H₅O₂ and 50 ml HNO₃ was used for etching the nickel interlayer.

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 reaction 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...
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 Ti2Ni 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 TiNi3 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+Ti2Ni. The bright area corresponds to the Ti2Ni 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 λ+χ+αFe phases [20,21]. As reported by Kundu and Chatterjee [23,24] the nickel concentration in this area is too low to form any nickel based intermetallic compound. The brightest layer has been recognized as a combination of FeTi. 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 steel 195 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$_2$Ni, TiNi and TiNi$_3$ intermetallic phases. The hardness values for those phases were as follow: 446 HV for Ti$_2$Ni, 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.

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
1001 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 Ti2Ni, TiNi, TiNi3 were observed at the titanium nickel side of the diffusion joint, however the TiNi, and TiNi3 phases were not found at 1000°C. There where only islands of αTi + Ti2Ni in the Ti2Ni matrix. The thicknesses of the Ti2Ni, TiNi and TiNi3 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 γFe + Ni appears between nickel and stainless steel. The mixture of λ + FeTi + Ti, FeTi + Ti, λ + αFe and λ + χ + αFe 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