Interaction between Al 99.5 and stainless steel at elevated temperature and pressure

Interakcija med Al 99,5 in nerjavnim jeklom pri povišani temperaturi in tlaku

Abstract

The aim of this work was to investigate interaction between aluminium 99.5 and austenitic stainless steel 1.4301 or ferritic stainless steel 1.4767 at elevated temperature and pressure. Three series of tests were done. Samples were pressed with Thermomechanical simulator of metallurgical states "Gleeble 1500D". Before each series of bonding, aluminium 99.5 was cleaned in a liquid agent for degreasing and removal of oxides "Nabadur 152 (5 %)". After pressing, the samples were prepared for metallographic analysis. Samples were analyzed under an optical or a scanning electron microscope.

Cleaning agent Nabadur 152 (5 %) ensures proper cleanliness of the surface of the aluminium, the steel is not needed to be cleaned. The formation of the joint is heavily dependent on the geometry of the tool. In order to achieve a good bond, bonding at a temperature of 550 °C or more is required. The holding time at the temperature for successful bonding should be at least 5 s. Minimum force required for the formation of the bond is 13 kN.

Key words: bonding, aluminium alloys, stainless steel, elevated temperature, elevated pressure
Introduction

Diffusion bonding of materials in the solid state is a process for making a monolithic joint with the formation of bonds at an atomic level as a result of joining the opposite surfaces due to local plastic deformation at elevated temperatures, which increases the interdiffusion at the surface layer of the materials, which are brought together.[1]

Bonding in solid state is a joint mark for proceedings in which there is no bonding material, but the material is heated only to the pitting state and the bond is achieved by pressing. Here the surface is brought to the atomic level distance (distance between the atoms), therefore the adhesion forces influence on the bond. Due to the increase in temperature at the boundary layer the process of diffusion occurs. Because the bond is deformed due to the pressure, a combination with the high temperature processes of recrystallization occurs.[2]

Prior to the joining of aluminium and stainless steel it is necessary to both materials are adequately prepared. It is very important that the surface is clean and of suitable roughness, because only in this way a good bond can be achieved. Stainless steel and aluminium have different melting temperature, so these materials must be bonded under the melting point of aluminium. Temperatures higher than 550 °C are problematic, because there is a softening of aluminium. For joining of steel and aluminium by rolling, the aluminium is heated up to a temperature of 450–550 °C, while the steel is heated to 400 °C. It is very important, that the oxides do not form on the surface, which would prevent the bonding. Problems at the bonding with the rolling process may be mainly caused by oxides in the steel, which are not disrupted, while the oxides of the aluminium may be disturbed easily. [3] During the bonding brittle intermetallic phase may also be formed, which weakens the joint. [4] The formation of this phase takes place at temperatures above 500 °C. [5] The thickness of the intermetallic phase can be from a few nm to a few hundred μm. The most common phase resulting from the bond between the stainless steel and aluminium are phases from the ternary system Fe-Cr-Al. [6] To achieve the best possible bond, it is important that the intermetallic phase is as narrow as possible or that they are not present. [4] Acceptable thickness of the intermetallic layer, which does not impact the strength of the bond is 3–5 μm, while the thickness exceeding 10 μm causes the bond unusable.[7]

The bonding speed is also important, which vary from procedure to procedure. Speeds may range from a few cm/s and up to 1600 m/s at the explosion bonding. To achieve good joints between aluminium and steel at least 50 % deformation is required. The bonding time varies from a few hundredths of a second and up to several hours, and are dependent on the bonding process and other parameters, such as speed, temperature, pressure and atmosphere. The aim of this work was to investigate the interaction between aluminium 99.5 and austenitic stainless steel 1.4301 or ferritic stainless steel 1.4767 at elevated temperature and pressure.

The company Talum, d. d., wanted to produce new products which would be produced by bonding in the solid. The purpose of the study was to investigate the interaction between 99.5 aluminium and stainless steel, as well as to establish optimal conditions for achieving a good bond. To achieve a good bond it is very important the surface preparation, which has to be properly cleaned. All impurities must be removed, such as oxides, dust and moisture.

Experimental work

With the aim of bonding of aluminium 99.5 (AA1050, Table 1, soft annealed state) and austenitic stainless steel 1.4301 or ferritic stainless steel 1.4767 at elevated temperature and pressure, three different series of compression tests were made at various experimental conditions, presented in Table 2. Tests of bonding were carried out by Thermomechanical simulator of metallurgical states Gleeble 1500D. Bonding was carried out at different temperatures where the samples were induction heated to the testing temperature, holding at the coupling temperature times, the compression speeds and displacements of the tool on the sample surface of approximately 2 cm². Samples were subsequently analyzed by optical
(Olympus BX61) and scanning electron microscopy (Jeol 5610). Using a computer simulation program Thermo-Calc, based on different databases, a simulation of thermodynamic equilibrium bond formed between the steel and aluminium was produced.

In a first series 7 samples were compressed. Bonding was carried out at a temperature of 550 °C and 600 °C, movement of the tool was 0.4 mm, while the retention time at the temperature of compression was 0 s and 10 s. Samples 1.2, 1.5 and 1.6 were analysed using microscope. In the first two samples, a ferrite stainless steel 1.4767 and aluminium 99.5 were used, and in the third sample austenitic stainless steel 1.4301 was used. All three samples were compressed at a temperature of 550 °C, samples 1.5 and 1.6 were maintained at a temperature of compression for 10 s, while the sample 1.2 was only compressed. Formation of the bond was observed in samples 1.5 and 1.6, whereas at the sample 1.2 the bond was not achieved. At the samples where the bond was formed an intermediate layer was observed, whereas the thickness in the sample 1.5 was 4.08 µm and in the sample 1.6 5.84 µm. In both cases, the samples were compressed with a force of 13 kN. At the same time 59 % and 40.55 % deformation of aluminium was achieved.

In the second series 6 samples were compressed, using profiled tool. Bonding was carried out at a temperature of 560 °C. The displacement of the tool was set on 0.4 mm, tool speed was 100 mm/s, the holding times were (0, 2, 4, 6 and 8) s. From this series, the samples 2.1, 2.2, 2.3 and 2.4 were analyzed. In all samples aluminium 99.5 and ferritic stainless

<table>
<thead>
<tr>
<th>Sample</th>
<th>Upper sample</th>
<th>Lower sample</th>
<th>Temperature (°C)</th>
<th>Movement (mm)</th>
<th>Holding time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>550</td>
<td>0.4</td>
<td>-</td>
</tr>
<tr>
<td>1.2</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>550</td>
<td>0.4</td>
<td>-</td>
</tr>
<tr>
<td>1.3</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>550</td>
<td>0.4</td>
<td>10</td>
</tr>
<tr>
<td>1.4</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>600</td>
<td>0.4</td>
<td>-</td>
</tr>
<tr>
<td>1.5</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>550</td>
<td>0.4</td>
<td>10</td>
</tr>
<tr>
<td>1.6</td>
<td>Al 99.5</td>
<td>1.4301</td>
<td>550</td>
<td>0.4</td>
<td>10</td>
</tr>
<tr>
<td>1.7</td>
<td>Al 99.5</td>
<td>1.4301</td>
<td>600</td>
<td>0.4</td>
<td>10</td>
</tr>
<tr>
<td>2.1</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>560</td>
<td>0.4</td>
<td>2</td>
</tr>
<tr>
<td>2.2</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>560</td>
<td>0.4</td>
<td>6</td>
</tr>
<tr>
<td>2.3</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>560</td>
<td>0.4</td>
<td>8</td>
</tr>
<tr>
<td>2.4</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>560</td>
<td>0.4</td>
<td>4</td>
</tr>
<tr>
<td>2.5</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>560</td>
<td>0.4</td>
<td>0</td>
</tr>
<tr>
<td>2.6</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>560</td>
<td>0.4</td>
<td>2</td>
</tr>
<tr>
<td>3.1</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>540</td>
<td>0.4</td>
<td>5</td>
</tr>
<tr>
<td>3.2</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>540</td>
<td>0.4</td>
<td>5</td>
</tr>
<tr>
<td>3.3</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>540</td>
<td>/</td>
<td>5</td>
</tr>
<tr>
<td>3.4</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>540</td>
<td>0.6</td>
<td>5</td>
</tr>
<tr>
<td>3.5</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>540</td>
<td>0.6</td>
<td>3</td>
</tr>
<tr>
<td>3.6</td>
<td>Al 99.5</td>
<td>1.4767</td>
<td>540</td>
<td>0.6</td>
<td>8</td>
</tr>
</tbody>
</table>

**Table 1**: Chemical composition of investigated Al-alloy Al99.5 (AA1050) [1]

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA1050</td>
<td>0.07</td>
<td>0.26</td>
<td>&lt;0.001</td>
<td>-</td>
<td>&lt;0.001</td>
<td>-</td>
<td>&lt;0.002</td>
<td>-</td>
<td>99.50</td>
</tr>
</tbody>
</table>

**Table 2**: Experimental samples
Steel 1.4767 were used. At these conditions the bond in all sample was not achieved. In this series of compression test the compression force was ranging from 8 kN to 20.5 kN, while the deformation of the aluminium was in the range between 30 % and 60 %.

In the third series 6 samples were compressed (aluminium 99.5 and ferritic stainless steel 1.4767). Bonding was carried out with a profiled tool at a temperature of 540 °C. The displacement of the tool varied from 0.4 mm to 0.6 mm. The holding time at the temperature of compression (3 s, 5 s and 8 s) also varied. In this series, samples 3.3, 3.4 and 3.6 were thoroughly analyzed. Bond occurred only in the sample 3.6. In this sample, a force of 16 kN, and deformation of the aluminium 55.46 % were achieved. When the bond occurred, 1 µm thick discontinuous intermediate layer appeared, whose composition corresponds to phases AlCr<sub>2</sub> and Al<sub>8</sub>Cr.

Results and discussion

First series

Using optical microscopy samples 1.2, 1.5 and 1.6 were analyzed. The bond was achieved in samples 1.5 and 1.6. Therefore, in these two samples the analysis with a scanning electron microscope was made.

Sample 1.2 was heated up to 550 °C, where it was held for 5 s, so the temperature was constant over the entire sample. Then it was compressed with a force of 15 kN. The displacement was set to 0.4 mm. At the compression of the sample 1.2 there is a 43.2 % deformation of the aluminium, while no deformation of the steel occurred. The different width of the gap is due to non-parallel jaws and the tool during pressing.

Here, the sample 1.5 was heated to the temperature of 550 °C, where it was held for 5 s, so the temperature was constant over the entire sample. Then it was compressed with a force of 15 kN. The displacement was set to 0.4 mm. At the compression of the sample 1.2 there is a 43.2 % deformation of the aluminium, while no deformation of the steel occurred. The different width of the gap is due to non-parallel jaws and the tool during pressing.

Here, the sample 1.5 was heated to the temperature of 550 °C, where it was held for 5 s, so the temperature was constant over the entire sample. Then it was compressed with a force of 15 kN. The displacement was set to 0.4 mm. At the compression of the sample 1.2 there is a 43.2 % deformation of the aluminium, while no deformation of the steel occurred. The different width of the gap is due to non-parallel jaws and the tool during pressing.

Here, the sample 1.5 was heated to the temperature of 550 °C, where it was held for 5 s, so the temperature was constant over the entire sample. Then it was compressed with a force of 15 kN. The displacement was set to 0.4 mm. At the compression of the sample 1.2 there is a 43.2 % deformation of the aluminium, while no deformation of the steel occurred. The different width of the gap is due to non-parallel jaws and the tool during pressing.

In Figure 1 is presented the left side of the compressed sample, where the displacement of aluminium occurred. The thickness of the aluminium in this part was 456.15 µm, the steel was 710.13 µm. As illustrated in Figures 4b–d, the thickness of the steel was 706.30 µm or 710 µm at the compressed part of the sample, while the thickness of the aluminium was 445.84 µm or 474.27 µm. Figures 4e and 4f show the thickness of the steel and aluminium on the uncompressed part of the sample.
thickness of the steel was 706.75 µm, while aluminium was 748.84 µm. At the sample 1.6 40.55 % deformation of the aluminium occurred, and the same as at the sample 1.2 and 1.5, no deformation of the steel occurred. Sample 1.6 was previewed under the scanning electron microscope. Thickness and chemical composition of the intermediate layer was determined, resulting in the compression test of the sample 1.6. In Figure 5 presents the areas where the surface analysis of the sample 1.6 was done.

Figure 1: Thickness of the sample 1.5 after compression test: left compressed part (a), right compressed part (b), middle compressed part (c) and left uncompressed part (d).

Figure 2: EDS surface analysis of bonded part of the sample 1.5.
From Figure 5 it can be seen that the area 1, which presents the steel, contains amount fractions 0.750 % Si, 19.734 % Cr, 1.537 % Mn, 70.471 % Fe, 7.298 % Ni and 0.210 % Mo. Section 2 (intermediate layer) contains amount fractions 67.847 % Al, 0.284 % Si, 5.955 % Cr, 0.445 % Mn, 23.326 % Fe and 2.143 % Ni, while section 3 (aluminium) contains 99.454 % Al, 0.333 % Cr and 0.213 % Fe. The thickness of the intermediate layer that forms between the bonding of aluminium and steel was also measured and it was 5.84 µm (Figure 6).

Using the Thermo-Calc programme, isoplete equilibrium phase diagram (Figure 7) for the intermediate phase was constructed, resulting in a sample 1.6. From the Figure 7 can be seen that when the mass concentration of Fe is 36.174 %, following phases are possible: Al$_{8}$Cr$_{5}$ and Al$_{13}$Fe$_{4}$.

**Second series**

In the second series, where 6 compressing tests were made, profiled tool was used. In all sample no bond occurred.

**Third series**

In the third series 6 samples were compressed under certain conditions. Using an optical microscope, samples 3.3, 3.4 and 3.6 were analyzed. The bond occurred only at the sample 3.6, which was further analyzed using a scanning electron microscope. Sample 3.3 was heated to a temperature 540 °C, where it was held for 5 s so the temperature was homogeneous throughout the sample. This was followed by compressing the sample with a force of 23 kN.

The displacement of the jaw was 0.4 mm. On Figure 8a it can be seen the part of a sample 3.3, which was compressed on the rib of the tool. The thickness of the aluminium in this part of the sample was 198 µm, thickness of the steel was 468 µm. Figures 8b and 8c show a part of the compressed sample, which was in the ‘valley’ of the tool. The thickness of aluminium on this part was 892 µm (Figure 8b), while the thickness of the steel was 463 µm (Figure 8c).

Sample 3.4 was heated to 540 °C where it was held for 5 s so the temperature was uniform throughout the sample. This was followed by pressing with a force of 22.5 kN. Rear calipers were set on 0.4 mm. Figure 9a shows a part of a sample 3.4, which was compressed by a rib of the tool. The thickness of the aluminium was 372 µm and the steel 464 µm. Figures 9b and 9c present the location where the material was compressed by a groove of the tool. Here the thickness of aluminium was 829 µm (Figure 9b), the thickness of the steel was 467 µm (Figure 9c).

Sample 3.6 was first heated to a temperature of 540 °C. At this temperature was held for 5 s and then pressed with a force of 16 kN, the displacement of the jaws was set to 0.6 mm. Figure 10 shows the bond between aluminium and steel in the sample 3.6.

In Figures 11a, b and c the thickness of aluminium and steel in specific parts of the sample can be seen. Figure 11a represents a part of the sample, which was compressed with a rib of the tool, Figures 11b and c represent a part of the sample, which is in the groove of the tool. Figure 11a shows that the thickness of the aluminium was in this case 334 µm and steel 466 µm. In Figure 11b the measured thickness of the aluminium was 815 µm, the thickness of the steel, measured in Figure 11c was 464 µm.

As the third series of compression tests lead to the bond only in the sample 3.6, it was further analyzed using a scanning electron microscope. In the sample the bond without an intermediate layer (Figure 12a) or with an intermediate layer (Figure 12b) was obtained, which was not continuous.

At the bond, without the intermediate layer surface and line analysis was made. The results of both analysis are shown in Figures 13 and 14.
From the results of the surface analysis shown in Figure 13 can be seen that the area 1 (aluminium) contains amount fractions 99.538 % Al, 0.003 % Si, 0.066 % Cr and 0.393 % Fe. Region 2, which represents the steel contains amount fractions 5.627 % Al, 0.224 % Si, 17.574 % Cr and 76.575 % Fe.

Results of surface analysis in Figure 14 show, that the area where an intermediate layer between aluminium and steel (section 1) was formed, contains amount fractions 45.569 % Al, 0.096 % Si, 10.134 % Cr and 44.201 % Fe. Region 2, which presents aluminium contains 99.378 % Al, 0.120 % Cr and 0.501 % Fe.

**Figure 4:** Thickness of the sample 1.6: left compressed part (a) and (b), right compressed part (c), middle compressed part (d) and right uncompressed part (e) and (f).
The steel (section 3) contains amount fractions 6.079 % Al, 0.402 % Si, 17.204 % Cr and 76.315 % Fe.

Using Thermo-Calc program isoplete equilibri um phase diagram (Figure 15) for the intermediate layer was constructed, resulting in a sample of 3.6. From the diagram it can be seen that at the mass concentration of 58.4 % Fe, and 12.5 % Cr, phases AlCr2 and Al8Cr5 may occur. In all three series it was observed that the jaws on the Thermomechanical simulator of met-

allurgical stages Gleeble 1500 D are not completely parallel resulting in uneven bonds. On bonding a profiled tool had great impact. Due to the non-parallel jaw on which a tool for compression is placed, the force on certain points of the material is greater than elsewhere. This resulted in a different filling of the grooves of tools with aluminium. For the determination of the quality of the bond, further tests should be made such as bending tests or quantitative tensile tests of double-layered sheet.

**Figure 5:** EDS surface analysis of bonded part of the sample 1.6.

**Figure 6:** The thickness of intermediate layer in sample 1.6.

**Figure 7:** Equilibrium isoplete phase diagram Al-Fe-Cr-Ni-Mn-Si.
Figure 8: The thickness of the material in the sample 3.3: rib of the tool (a) and in the ‘valley’ of the tool – aluminium (b) and steel (c).

Figure 9: The thickness of the material in the sample 3.4: rib of the tool (a) and in the ‘valley’ of the tool – aluminium (b) and steel (c).
Figure 10: Bond in the sample 3.6.

Figure 11: The thickness of the material in the sample 3.6: rib of the tool (a) and in the valley of the tool – aluminium (b) and steel (c).
Interaction between Al 99.5 and stainless steel at elevated temperature and pressure

Figure 12: The bond between Al 99.5 and ferrite stainless steel 1.4767 of the sample 3.6: there is no intermediate layer between the aluminium and steel (a) and discontinuous intermediate layer between the aluminium and steel (b).

Figure 13: EDS surface analysis of sample 3.6.

Figure 14: EDS surface analysis of sample 3.6, where the intermediate layer occurred.
Conclusions

According to the results, it can be concluded:

— To achieve a good bond, the aluminium surface must be properly cleaned. Cleaning agent Nabadur 152 (5 %) ensures proper cleanliness of the surface of the aluminium so that the steel is not needed to be cleaned.

— The formation of the bond is heavily dependent on the geometry of the tool. The best results showed unprofiled tool. In the first series, where unprofiled tool was used two good bonds along the entire length of the sample were achieved. When the profiled tool was used, the bond only on a small area of the sample was achieved. Profiled tool should have shallower grooves and broad ribs.

— It is important to ensure parallelism of the jaw. Non-parallelism of the jaws causes a local increase in the forces leading to the formation of gaps and uneven filling of the grooves in the tools.

— In order to achieve the bond it is required compressing at a temperature of 550 °C or more. The time for holding the temperature for bonding should be at least 5 seconds.

— Minimum force required for the formation of the bond is 13 kN.

— The minimum deformation of the material, which is necessary for the formation of the connection, is 40.6 %.

— At the formation of the bond, intermediate layer was formed. The thickness of this layer depends on the holding time at the bonding temperature and the type of stainless steel. In the case of ferritic stainless steel with the same bonding conditions, a thinner intermediate layer than in austenitic stainless steel was obtained. The bond with the intermediate layer of 1 μm thick formed at a temperature of 540 °C and the holding time for 8 s. The composition of the intermediate layer is based on the ternary system Fe-Cr-Al. At the bonding of Al99.5 with ferrite or austenitic stainless steel the occurrence of the following phases is possible: Al₉Fe₂, Al₁₃Fe₄, Al₁₃Cr₂, Al₁₃Fe₄, Al₁₃Cr₂, and Al₈Cr₅.

References


