Investigations on Corrosion Properties of Ultrasound-Enhanced Friction-Stir-Welded Aluminum/Dual-Phase Steel Joints

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Dedicated to Prof. Dr.-Ing. Wolfgang Bleck on the occasion of his 70th birthday.

Friction stir welding (FSW) is a solid-state joining method that is suitable for joining dissimilar materials such as aluminum and steel due to its comparatively low process temperatures. Such hybrid joints are of great interest in view of lightweight construction efforts in various industrial sectors such as transportation. As the combination of different metals in hybrid structures may cause corrosion problems in the welding area because of the formation of a galvanic couple, the corrosion properties are investigated. This work also deals with the influence of additionally transmitted power ultrasound during friction stir welding on the joint properties of AA6061/DP600. Light microscopic analysis and radiographic results show differences in the amount and size of steel particles in the near-surface area of the joints depending on the used ultrasound power. Although the aluminum alloy and the dual-phase steel exhibit a Volta potential difference of about 0.8 V in scanning Kelvin probe (SKP) measurements, the measured corrosion current densities on different positions of the AA6061/DP600 joints in 0.5 M sodium chloride solution are only low and no enhanced Galvanic corrosion is observed. A distinct influence of the power ultrasound on the corrosion properties is not given.

1. Introduction

The substitution of steel by aluminum offers great potential for several industrial fields, such as the mobility sector.[1] Apart from possible weight reductions, also, design aspects such as the shift of the vehicle’s center of gravity can have a beneficial impact.[2] In this context, firmly bonded joining methods achieve the highest joint strengths. Nevertheless, conventional fusion welding processes are not suitable for producing high-quality aluminum/steel joints due to an excessive formation of detrimental brittle intermetallic phases (IMPs) such as the aluminum-rich phases FeAl3 and Fe2Al5.[3–5] Solid-state joining processes such as friction stir welding (FSW), in contrast, operate at a comparatively low process temperature of only about 80% of the melting temperature and thus drastically reduce the formation of such IMPs.[6] The process of FSW can be subdivided into the four steps of plunging, dwelling, welding, and extraction. The plunging is described by the defined penetration of a rotating cylindrical tool with a probe at the end into the joining partners. Due to the development of frictional heat between the probe and the material, a locally plasticized zone develops and ensures the plunging of the probe. The subsequent dwelling enables a steady state of the plasticized zone. By moving the cylindrical tool along the respective faying surface, the plasticized materials get stirred and a firmly bonded joint is achieved. After retracting the tool, an end hole remains, which can be avoided by different strategies.[1] Using this FSW method, a joint efficiency of about 90% could be already achieved by Wang et al. for dissimilar AA5083–H116/HSLA–65 joints.[7] As mentioned before, IMPs cannot be avoided completely in joining such dissimilar metals by the FSW process, because they play a major role in the resulting bonding mechanisms. Uzun et al. showed a reduction of the fatigue properties on aluminum/steel joints associated with the diffusion of aluminum into steel at the interface.[8] The investigations of Watanabe et al. on friction stir spot welded dissimilar aluminum/steel joints proved the presence of compound layers of Fe4Al13 and Fe2Al5 and showed a
correlation between dwell time and IMP thickness.[9] In the work of Tanaka et al. the formation of a brittle IMP on aluminum/steel joints was directly addressed with regard to an inconstant temperature-controlled two-step formation process of such layers.[10] Therefore, a very important fact is the controlled development of intermetallic phases, which can be realized using optimal process parameters or, if an FSW machine is reaching its limits, using hybrid joining processes such as ultrasound-enhanced friction stir welding (USE-FSW). This method is characterized by the additional transmission of power ultrasound into one of the joining partners. The ultrasound enhancement has two cooperating mechanistic modes of action. The acousto-plastic effect was discovered by Blaha and Langenecker in 1955.[11] They proved a reduction of the yield stress under the influence of high-frequency power ultrasound, which is beneficial for FSW with ultrasound support. Therewith, the work of Park showed a reduction of the process forces.[12] Zhong et al. as well as Straß et al. also detected an enhanced stirring for the ultrasound enhancement during FSW.[13,14] In addition, Liu et al. found the possibility to increase the welding speed by ultrasound support.[15] The second mechanistic mode of the power ultrasound is the interaction with irregularities such as oxide layers and IMPS. Jene proved a reduction of oxide layers for similar aluminum/aluminum joints when applying power ultrasound during FSW,[16] whereas Straß as well as Thomä found thinner continuous intermetallic layers for dissimilar aluminum/magnesium and aluminum/steel joints.[17,18] These advantageous properties qualify USE-FSW as an innovative joining method for similar and dissimilar joints. Regarding the literature, there are only a few publications relating to the influence of additional power ultrasound on the corrosion properties of the resulting dissimilar joints. The combination of different metals in hybrid structures may cause corrosion problems in the welding area because of the formation of a galvanic couple, which leads to severe corrosion of the less noble element. The improved stirring of dissimilar aluminum and magnesium cast alloys by ultrasound enhancement resulted in a more pronounced corrosive attack of the nugget, whereas the application of the USE-FSW process on AA6061/SAE1006 joints showed no significant changes in the corrosion properties.[19–22] In addition, an accelerated corrosion of the magnesium alloy in a chloride-containing electrolyte was found when it was measured in contact with the nugget area.[19–21]

The current work investigates the influence of ultrasound with varying power input on the corrosion properties of dissimilar AA6061/DP600 joints in comparison to the conventional FSW process and how they are correlated with the microstructure of the joints. The corrosion properties of the hybrid joints were investigated by scanning Kelvin probe (SKP) measurements in humid air and electrochemical measurements (open circuit potential and potentiodynamic polarization) in sodium chloride solutions. The results related to the microstructure and the mechanical properties have already been presented in an earlier (open access) publication—together with investigations on aluminum/magnesium hybrid joints.[19] However, because the results of the corrosion investigations cannot be discussed without the microstructural investigations, some essential results are briefly included here.

2. Experimental Section

2.1. Materials

For the aluminum/dual-phase steel joints, the aluminum wrought alloy AA6061 (AlMg1SiCu) and the dual-phase steel DP600 (1.0936) were used. Their geometry was 280 mm × 100 mm × 3 mm. Their chemical compositions according to the specifications of the manufacturers are listed in Table 1.

2.2. Processing of the Joints

The aluminum/dual-phase steel joints were realized on a modified four-axis universal machining center DMU80T from DMG Mori in a butt joint configuration under force control. The dual-phase steel was placed on the advancing side and the aluminum plate on the retreating side of the welding direction. Furthermore, the welding tool consisted of a tungsten-based alloy with 1% lanthanum oxides. Optimal FSW process parameters were identified for 2.5 kN axial force, 1250 rpm rotational speed, 30 mm min⁻¹ feed rate, a 2° tilt angle, and a lateral offset of the tool center axis of 3 mm into the aluminum. Therefore, no direct contact between the probe and the steel should occur. For the USE-FSW process, the machine was additionally equipped with an ultrasonic roll seam module of Schunk. It ran synchronously and parallel to the FSW tool in the process and transmitted the power ultrasound with a resonance frequency of 20 kHz, a generator maximum power of 3000 W, and a maximum amplitude of 38 μm. Within the scope of the investigations, the ultrasound was introduced from the steel side and the power was varied in two steps of 225 and 1000 W. A more detailed description of the experimental setup is given in an earlier publication.[19]

2.3. Investigation of Microstructure and Joint Quality

The preparation of the AA6061/DP600 joint samples for microscopic analysis of the cross-section took place by cutting the material compounds of aluminum/steel and embedding them with cold embedding resin. Afterward the samples were ground with wet silicon carbide paper (up to P4000), then polished with a Diapora Nan 1 µm suspension, and finally cleaned and dried with ethanol. Light microscopic investigations were conducted on a light microscope Olympus GX51. Radiographs of the joints were performed using an X-ray system, which is an in-house development of Fraunhofer IZFP, equipped with a 225 kV microfocus X-ray tube, a 4 megapixel detector, and a 16 bit ADC. The

Table 1. Chemical composition of the investigated materials.

| Material | Elements [wt%] |
|----------|---------------|
| AA6061   | Si  Fe  Cu  Mn  Mg  Cr  Zn  Ti  Al |
|          | 0.64 0.51 0.21 0.14 0.89 0.15 0.04 0.05 Bal. |
| DP600    | C  Si  Mn  P  S  Al  B  N  Fe |
|          | 0.14 1.5 2 0.07 0.015 0.015 0.005 0.009 Bal. |
radiographic images were achieved by a tube voltage of 100 kV, a tube current of 0.3 mA, and an exposure time of 30 s. Again, a more extensive description regarding this experimental setup is given in the former publication.

### 2.4. Corrosion Investigations

The surface of the samples was ground with wet silicon carbide paper (up to P1200), rinsed with ethanol, and dried prior to all measurements to achieve comparable conditions for all samples.

To obtain spatially resolved information about local potential differences on materials' surfaces the SKP is a suitable method. For the investigations herein of Volta potential differences between the aluminum, the dual-phase steel, and possibly generated intermetallic phases after the FSW and USE-FSW process an SKP setup with height control (SKP_KM, Wicinski-Wicinski GbR) was used. The measuring probe was a chromium-nickel wire with a diameter of 100 μm. The probe was calibrated at measurement conditions (humid air >90% RH) with a copper/copper sulfate reference electrode. The measured potential values are given with respect to the standard hydrogen electrode (SHE). The scanned surface area was 25 × 5 mm² and the step size was 50 μm in X- and Y-direction.

Electrochemical measurements were conducted for the investigation of the corrosion properties of the base materials and joints. They were conducted in 0.5 M sodium chloride solution. Figure 1 shows the three-electrode setup within a minicell (measurement area 0.071 cm²) that was used to enable measurements on different areas of the joints. The areas are the aluminum nugget area (stirred zone), the aluminum nugget/dual-phase steel transitional area, and two positions about 3 cm from the weld. These positions, one on the aluminum and one on the dual-phase steel side, represent the properties of the base materials. The investigated samples were used as working electrode, a platinum electrode as counter electrode, and a standard calomel electrode (+245 mV vs SHE) as reference electrode. The recorded values were converted to values against the SHE.

The open circuit potential measurements and the potentiodynamic polarization were done with a Zahner IM 6 measurement unit. The open circuit potential was recorded for 60 min prior to the polarization experiment. The potentiodynamic curves were started at −0.05 V relative to the open circuit potential (Eoc) and recorded up to +0.3 V relative to Eoc with a scan rate of 1 mV s⁻¹.

### 3. Results and Discussion

#### 3.1. Microstructure and Joint Quality

##### 3.1.1. Light Microscopic Investigations

The influence of the different joining conditions (FSW and USE-FSW with varying ultrasound power) on the microstructure of the AA6061/DP600 joints was investigated in detail by light as well as scanning electron microscopic analysis regarding the shape of the interface, the development of hooks and IMPs, as well as particle size and volume fraction in Thomä et al.[19] Regarding the present corrosion investigations, light microscopic analysis was focused on the near-surface area of the stir zone (Figure 2).

A comparison of the areas near the surface of the stir zone showed a lower amount of steel particles as well as a smaller particle size with increasing ultrasound power. These results are in accordance with investigations on other aluminum/steel hybrid joints.[23,24]

##### 3.1.2. Radiography (X-ray)

As described by Thomä et al. the occurrence, size, and distribution of foreign particles that may affect the corrosion behavior of the welding area can be identified by radiographic measurements.[19] The X-ray images in Figure 3 display a significant influence of the applied ultrasound power on the occurrence, quantity, and size of such foreign particles. In addition to some pores, the conventional FSW joint reveals numerous particles on the aluminum side of the joining area, which are finely distributed along the entire length of the weld (Figure 3a). The ultrasound-enhanced FSW process with an ultrasound power of 225 W leads to less, but bigger particles, which only appear along the first half of the weld length (Figure 3b). Increasing the ultrasound power up to 1000 W...
results in avoidance of foreign particles along the complete weld seam (Figure 3c). These findings are in accordance with the results on AA6061/SAE1045 joints and AA6061/SAE1006 joints.\cite{23,25}

Figure 2. a) Scheme of the general position of light microscopic cross-section images of AA6061/DP600 joints as well as detailed images for b) FSW, c) USE-FSW with 225 W, and d) USE-FSW with 1000 W.

Figure 3. Radiographs (X-ray) of AA6061/DP600 a) FSW joint, b) USE-FSW joint with 225 W ultrasound power, and c) USE-FSW joint with 1000 W ultrasound power. Reproduced under the terms of the CC-BY license.\cite{19} Copyright 2019, Thomä et al., Published by Wiley-VCH.
3.2. Corrosion Investigations

3.2.1. SKP Measurements

Figure 4 shows the measured Volta potentials of the welding zones of the AA6061/DP600 FSW joint (a) and the USE-FSW joints with 225 W (b) and 1000 W (c) ultrasound power as 2D color plots. The recording of the whole plot of one sample takes about 16 h. Therefore, the plots illustrate not only the Volta potential differences between the different materials and areas, but also their oxidation properties in humid air during the measurement time. At the beginning of the measurements, the Volta potential difference between the AA6061 base alloy (0 to about 5 mm on x-axis) and the DP600 dual-phase steel (20 to 25 mm on x-axis) is about 0.75 to 0.9 V. Due to surface oxidation processes in humid air, the Volta potentials shift to more positive values during the measurement. As both materials exhibit a similar shift of about 0.2–0.25 V, the Volta potential difference between the materials keeps nearly constant, whereas the aluminum alloy shows negative values (−0.4 to −0.5 V) and the steel positive values (+0.3–0.4 V). This behavior is independent of the processing conditions of the joints.

The nugget area (starting at about 7.5 mm on the x-axis) shows a changed oxidation behavior compared to the aluminum base alloy, which is visible in the potential plots of the FSW joint and the USE-FSW joint produced with an ultrasound power of 225 W (Figure 4a,b) after about 8–10 h of measurement. Although the aluminum base alloy has already reached constant Volta potential values, the potential of the nugget area shifts even further to more positive values. At the end of the measurements, the Volta potential difference between the aluminum base alloy and the nugget area is about 50–70 mV. Within the nugget area of the FSW joint (Figure 4a) and the USE-FSW joint produced with ultrasound power of 225 W (Figure 4b), some small spots of higher potential are visible, which may be attributed to steel particles in the nugget (spots are marked with black circles). These results are consistent with the radiographic measurements (Figure 3), which show foreign particles in the weld seams of these two joints but not for the joint processed with an ultrasound power of 1000 W. In the nugget area of that joint, no
potential spots are visible (Figure 4c) and the potential shift to more positive values of the nugget area compared to the aluminum base alloy is also not observed.

Between the aluminum nugget and the dual-phase steel—at the butt weld—there is a distinct potential step from the positive values of the DP600 steel (+0.3 V) to negative values of about −0.35 V, which are only about 0.1 V more positive than that of the AA6061 alloy. This is comparable to AA6061/SAE1045 joints.[25] A distinct influence of the used ultrasound power on the Volta potentials at the butt welds is not visible.

The SKP measurements result in about 0.75–0.9 V more negative Volta potential values for the AA6061 Al alloy compared to the DP600 dual-phase steel. This is a similar difference to that measured between AA6061 and the steels SAE1006 or SAE1045.[23,25] Hybrid joints between AC48000 aluminum alloy and magnesium alloys (AZ80 or AZ91) also showed a Volta potential difference of about 1 V, with the aluminum alloy exhibiting the more positive values in these cases.[19,20] Due to the measured distinct potential difference one could expect that the AA6061 alloy will show stronger corrosion in corrosive media than the steel, particularly if both phases are in simultaneous contact to an electrolyte; however, electrode kinetics and activation or passivation processes in an electrolyte may also play a decisive role.

3.2.2. Open Circuit Potential Measurements

Results of the SKP measurements in air are not directly transferable to the corrosion behavior in a liquid electrolyte, as a comparison of the potential values for the dual-phase steel and the aluminum alloy shows. During the SKP measurements in air, the Volta potential of DP600 steel was always about 0.8 V more positive than that of the AA6061 alloy. The values change significantly in the sodium chloride electrolyte (Figure 5). The OCP of the AA6061 alloy exhibits a shift from about −0.8 to −0.47 V within the first 5–10 min and then stabilizes around that value. These values are similar to the measured Volta potential differences in humid air and may be explained by passivation.

The OCP values of the DP600 dual-phase steel in the sodium chloride solution are much lower than the measured Volta potential differences in air. The positive values measured in air (Figure 4) change to only negative values in the electrolyte, starting at about −0.3 V shortly after the immersion and declining continuously to −0.45 V within the first 10–15 min and further to −0.51 V. This points to an activation of the steel leading to an OCP value that is more negative than that of the AA6061 Al alloy after 20–30 min. A similar behavior was observed for the steels SAE1006 and SAE1045,[23,25] but it is different to that of aluminum/magnesium hybrid joints, where an OCP difference of 0.9 V was measured between the base materials in the sodium chloride electrolyte similar to the Volta potential difference in air.[19,20,22]

The OCP curves of the nugget area are nearly identical to those of the AA6061 aluminum base alloy. This coincides with the SKP results, which show only minor potential differences between the nugget area and the AA6061 base material (Figure 4).

The OCP value of the aluminum nugget/steel transitional area shows nearly no changes during the measurement time. For the joints produced with ultrasound enhancement (Figure 5b,c) it starts at a value of about −0.47 V immediately after immersion in the electrolyte, which is the same value at which the aluminum base alloy and the nugget area stabilize after several minutes. Only the OCP value of the transitional area in the FSW joint (Figure 5a) shows a slight shift to more negative values with time.
However, this may also be an effect of the area ratio of aluminum/steel in the measured area because of the uneven weld seam and inaccurate positioning of the measurement cell and not an effect of the joining conditions.

Overall, no significant influence of the processing conditions of the aluminum/dual-phase steel joints on the OCP values can be detected. For all joints, the OCP difference between the materials is only about 40 mV or less after an immersion time of 60 min. Therefore, enhanced corrosion caused by a Galvanic element is not expected.

3.2.3. Potentiodynamic Polarization

Figure 6 shows the results of the potentiodynamic polarization measurements in 0.5 M NaCl solution on different positions of an AA6061/DP600 FSW hybrid joint (Figure 6a) and USE-FSW joints produced with 225 W (Figure 6b) and 1000 W (Figure 6c) ultrasound power. The measurements were started 60 min after immersion at open circuit potential.

The AA6061 aluminum alloy exhibits a free corrosion potential \(E_{oc}\) of around \(-0.45\) V and a pitting potential of around \(-0.42\) V, which is only slightly more positive than the free corrosion potential. The corrosion current density \(I_{corr}\) measured for the aluminum alloy is below 1 \(\mu\)A cm\(^{-2}\), indicating a low corrosion rate. At the pitting potential, the current density increases immediately up to values of several hundred microamperes per centimeter squared.

The free corrosion potential of the DP600 steel shows values between \(-0.51\) and \(-0.52\) V, which are about 70 mV more negative than that of the aluminum alloy. The polarization curve of the steel shows a continuous increase in the anodic region. The corrosion current density at the \(E_{oc}\) is significantly higher than that of the AA6061 aluminum alloy (4–6 \(\mu\)A cm\(^{-2}\)).

In case of the USE-FSW-joints (Figure 6b,c), the nugget area shows a free corrosion potential that is slightly cathodically shifted compared to the OCP measurements. The pitting potential is—with values of about \(-0.45\) V—also cathodically shifted compared to that of the aluminum base alloy. For all joints, the corrosion current density is somewhat higher than that of the AA6061 base alloy, indicating a slightly higher corrosion tendency. This behavior is different to that of earlier investigated aluminum/steel combinations with SAE1006 and SAE1045 steel, where the nugget area shows an anodic shift of the \(E_{oc}\) compared to the OCP measurements and corrosion current densities that are lower than that of the aluminum base alloy.\(^{[23,25]}\) The corrosion current density of the nugget area shows some influence of the processing conditions as the value on the FSW joint is higher than those on the USE-FSW joints (Figure 6). This may be attributed to the amount of steel particles within the measured area. The microscopic pictures (Figure 2) showed that the nugget areas of the welds contain steel particles and that the amount of particles is higher for the FSW joints compared to the USE-FSW joints. These particles appear to be responsible for the increased corrosion current densities compared to the aluminum base material, which corresponds to the slightly higher corrosion current densities of the steel.

The aluminum/steel transition region shows a similar behavior as the nugget area concerning a cathodically shifted free corrosion potential compared to the OCP as measured before. Thus, for all welded joints, the \(E_{oc}\) values are between the values of the DP600 dual-phase steel and the aluminum base alloy. The cathodic range of the polarization curves is comparable to that of
the DP600 steel. In the anodic range, the polarization curves first exhibit a continuous increase of the current density—comparable to the DP600 base material—and then a pitting potential and further progress comparable to that of the AA6061 aluminum base alloy. The $E_{oc}$ values of the aluminum/steel transition region show a shift to more positive values with increasing ultrasound power (Figure 6). This also leads to a shift of the pitting potential. One reason for this could also be the different proportion of particles in the nugget phase in this case. The layer of the IMP at the steel/aluminum interface, which becomes thicker with increasing ultrasonic power, may also play a role. [19]

However, it cannot be excluded that a slightly different aluminum-to-steel ratio within the measured area was present during the measurements. The corrosion current density is lower than that of the DP600 base material. This is a distinct difference to aluminum/magnesium hybrid joints where an increase of the corrosion current density of several orders of magnitude is observed at the magnesium/nugget transitional area.[19,20]

4. Conclusion

Friction stir welding was used to successfully produce AA6061/DP600 hybrid joints without and with additional power ultrasound (USE-FSW). The influence of the power ultrasound on the corrosion properties of the joints was investigated and correlated to the microstructure. The main findings are as follows: 1) The nugget phase of the welded joints contains numerous particle inclusions at the near-surface area of the stir zone. The optical microscopy and radiography studies prove that the size and number of particles depend on the power of the additionally used ultrasound. With increasing ultrasound power, fewer and smaller particles were observed. 2) The SKP measurements in humid air result in about 0.75–0.9 V more negative Volta potential values for the AA6061 aluminum alloy compared to the DP600 dual-phase steel. The nugget area shows a slightly different oxidation behavior compared to the aluminum base alloy. The observed potential spots in this region can be attributed to larger steel particles. 3) OCP measurements in 0.5 M sodium chloride solution show only a potential difference of 40 mV or less between the AA6061 aluminum alloy and the DP600 steel, with the steel now showing the more negative values. The potential shifts in the first minutes of exposure to the electrolyte reflect a passivation of the aluminum alloy in contrast to an activation of the steel. 4) The nugget area exhibits slightly higher corrosion current densities than the aluminum base alloy, which are attributed to the steel particles in this region. These will disturb the formation of a homogeneous passive layer and might be slightly activated, such as the steel substrate itself. 5) As a result of the low potential difference between the AA6061 and the DP600 in the sodium chloride solution, no enhanced Galvanic corrosion was observed for the aluminum/steel transitional area. The corrosion current density increases from the aluminum to the steel side, with the transitional area showing a corrosion current density in between the two base metals. 6) The corrosion properties of the joints are only slightly affected by the additional power ultrasound. The little lower corrosion current density in the nugget zone corresponds to a lower number and size of particle inclusions with the increasing of the ultrasound power.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

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