Effectiveness of Warm Water Treatment on Weld Bonding Performances of 6061 Aluminium Alloy

Norazwani Muhammad Zain1,a, Eida Nadirah Roslin2,b and Mohamad Asmidzam Ahamat3,c

1Fabrication and Joining Section, Universiti Kuala Lumpur Malaysia France Institute, 43650 Bandar Baru Bangi, Selangor, Malaysia.
2Automotive Engineering Section, Universiti Kuala Lumpur Malaysia France Institute, 43650 Bandar Baru Bangi, Selangor, Malaysia.
3Heating, Ventilating, Air Conditioning and Refrigerating Section, Universiti Kuala Lumpur Malaysia France Institute, 43650 Bandar Baru Bangi, Selangor, Malaysia.

Abstract
The positive impact of the surface pre-treatment on the adhesion strength has driven the idea to introduce this method in weld bonding process. This investigation is fundamentally about analyzing the effectiveness of warm water treatment on the weld bonded strength of Al 6061 alloy. There are two variables were used, i.e. temperature and treatment time. The Al 6061 surfaces have been immersed in warm water at three different temperatures (40, 50 and 60°C) with various treatment times specifically 15, 30 and 45 minutes. The substrates were then treated in the silane solution. The final stage of the work includes weld bonding process consists of green polyurethane adhesive bonding followed by resistance spot welding. The samples from all conditions were tested in a shear-tensile test according to EN ISO 14273 and microscopic analysis using Scanning Electron Microscope (SEM) to evaluate the bonding strength and mode of failures, respectively. The shear-tensile test results indicated that the samples that treated with warm water showed higher single lap shear strength compared to the non-treated sample. The weld bonded strength of Al 6061 is found to exhibit a remarkable dependence on warm water treatment. The temperature of warm water and treatment time had shown significant effects on the weld bonded strength of Al 6061.

Keywords: warm water treatment, weld bonding, adhesive bonding, resistance spot welding, polyurethane

INTRODUCTION
Weld bonding is one of innovative hybrid joining technology that has the benefits from the combination of resistance spot welding and adhesive bonding [1]. It integrates the physical force-based process of welding with the chemical force-based process of adhesive bonding [2]. In weld-bonded joints, both the spot weld and the adhesive layer contribute to the joint strength. The adhesive reduced significantly the stress level at the nugget root and the stress concentration at the edge of spot welds [3]. At the same time, the corrosion problem on the inner surface of the joint’s lap region has been successfully solved by weld bonding. The weld-bonded joints strength is greater and its reliability is promising compared to adhesive-bonded and spot weld-bonded joints alone [4]. Owing to the tremendous mechanical properties of weld-bonded joints, this joining process has been widely used in the production lines of automobiles and aviation fields.

The weld-bonded strength is determined by many factors, such as the dimension of the joints; the parameter of spot welding including welding current, time and electrode force; and the formulation of the adhesives. The properties of adhesives applied in weld-bonded technology have shown significant effects on fracture mode of the joints [5]. The preparation of the metal substrate prior to the weld bonding process is also important to ensure the excellent quality of weld-bonded joints. Tao et al. [6] and Boriwal [7] prepared the substrates by abrading with abrasive paper, degreasing with acetone and drying before weld bonding; to eradicate surface contamination and thus promote adhesion.

Stralin [8] was the first researcher who explored the surface treatment of aluminum alloys using boil water treatment in a way to enhance the epoxy adhesive bonding strength, followed Rider and Arnott [9]. The boil water treated aluminum alloys revealed the growth of hydrated oxide film on its surface contributed to the adhesion strength. Then, Underhill and Rider [10] approached with their findings on the lower temperature of water. They claimed that the hydrated oxide structure can be obtained at a temperature of 40°C. Moreover, previous effort by the author has sought to determine the effect of different surface treatment on the durability of polyurethane adhesive bonded aluminum alloy [11]. The result has shown that warm water (50°C) treatment followed by silanization has more significant effect on the durability of polyurethane adhesive bonding. The positive effects obtained previously have inspired us to continue research on the effectiveness of warm water treatment on the weld bonded strength of Al 6061 alloy. The goal of this work was to obtain a better understanding of the warm water treatment and other factors affecting the weld bonding strength, with a particular prominence on the role of the fractured surface condition of weld-bonded materials.
EXPERIMENTAL

Materials

Palm kernel oil based polyesteramide (PPKO) was supplied by Polymer Laboratory, Polymer Research Center, Universiti Kebangsaan Malaysia. Propylene carbonate (PC) was purchased from Sigma-Aldrich. 99% (3-Aminopropyl) triethoxysilane (ɣ-APS), ethyl alcohol 2, 4-diphenyl methylene diisocyanate (MDI) with an NCO content of 31% and bis(4-isocyanatocyclohexyl) methane (H12MDI) with an NCO content of 32% were supplied by Taat Bestari Sdn. Bhd.

Warm Water and Silane Treatment

The surface of Al 6061 substrates was rubbed with a clean tissue and immersed in acetone to remove grease and dirt. Then they were dipped in warm water at three different temperatures of 40, 50 and 60°C. The treatment time was also varied at 15, 30 and 45 min at each temperature. Then, the substrates were dried at ambient temperature. The warm water treated substrates were then immersed in 1% ɣ-APS solution for about 5 min to allow the surface silanization occurred. The substrates were dried at a temperature of 110°C for 5 min and the treated substrates were ready to be used in weld bonding process.

Polyurethane Adhesive Preparation

The polyurethane (PU) adhesive was prepared by reacting two types of isocyanate namely MDI and H12MDI with a ratio of 65:35 with the required amount of PPKO and other additives. The ratio of NCO: OH was 1.5. The mixture was mixed using a mechanical stirrer at 1000 rpm for about 10 seconds, and then the PU adhesive resin was ready to be applied.

Weld Bonding

The weld bonding process that used in this study was a Weld-Through method which is shown in Fig. 1. This method basically involved adhesive bonding followed by resistance spot welding (RSW) and it comprises four steps. In the first step, the PU adhesive was applied on the Al 6061 substrates followed by joint assembling. The joint were clamped to standardize the overlapped area and thickness of PU adhesive layer. The adhesive bonded joints were then cured in an oven at 120°C for 2 hours. Then, the RSW was carried out by using WIM Projection Spot Weld machine at room temperature as shown in Fig. 2. The welding electrode used in this study was RWMA Class II Chromium Copper alloy with the contact surface of 5.0 mm in diameter. The parameters used such as welding current, welding time and electrode force were 30kA, 4 cycles, and 4 kN, respectively. The non-treated sample of weld-bonded was labeled as WB-0 meanwhile for the warm water treated samples were labeled as in Table 1.

Table 1: Weld bonded sample labelling

<table>
<thead>
<tr>
<th>Sample Labelling</th>
<th>Warm Water Temperature (°C)</th>
<th>Treatment Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WB-0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>WB40-15</td>
<td>40</td>
<td>15</td>
</tr>
<tr>
<td>WB40-30</td>
<td>40</td>
<td>30</td>
</tr>
<tr>
<td>WB40-45</td>
<td>40</td>
<td>45</td>
</tr>
<tr>
<td>WB50-15</td>
<td>50</td>
<td>15</td>
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<td>30</td>
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<tr>
<td>WB50-45</td>
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<td>60</td>
<td>15</td>
</tr>
<tr>
<td>WB60-30</td>
<td>60</td>
<td>30</td>
</tr>
<tr>
<td>WB60-45</td>
<td>60</td>
<td>45</td>
</tr>
</tbody>
</table>

Figure 1: Weld bonding process by Weld-Through method.
Surface Roughness Test

In this study, topography images in 3D were generated by using Phenom Pro Suite software to determine the surface roughness, before and after warm water and silane treatment. The measurements were implemented in different areas, along four diverse directions as indicated in Fig. 3, to verify the treatment homogeneity and uniformity. The average roughness values, $R_a$, was then determined and recorded.

**Figure 3:** The directions of surface roughness measurement.

SEM-EDX Analysis

The substrate surfaces were characterized for microstructural by using Phenom World scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX). This surface analytical technique was also used to evaluate elemental analyses on the fractured surfaces plus to warm water treated surfaces.

Shear-Tensile Test

Universal Testing Machine model Instron armed with 250kN load cell was used to determine the shear-tensile strength of the weld-bonded joints. Five specimens of each sample were prepared in accordance with EN ISO 14273 standard and tested in tensile load. The dimension of the weld-bonded specimen is shown in Fig. 4. The crosshead speed was retained at 5 mm/min. The shear stress of weld-bonded joint was estimated by the formula: $\tau = F/A$, where $F$ is the maximum load and $A$ is the overlapped area of the joint. The overlapped area was fixed at 35 x 35 mm$^2$.

**Figure 4:** The dimension of the weld-bonded specimen.

RESULTS AND DISCUSSIONS

Influence of Warm Water Treatment on Surface Micro-Roughness

The effect of warm water treatment on surface micro-roughness is indicated in Table 2. Apparently, the average surface roughness of Al 6061 increases as the temperature of water and treatment time are increased. That means the Al surface become coarser after warm water treatment due to the oxide and hydrated oxide precipitated on the surface. This micro-roughness is expected to contribute to mechanical properties of weld-bonded Al 6061.

**Table 2:** Surface roughness of Al 6061 before and after warm water treatment

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average Roughness $R_a$ ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WB-0</td>
<td>1.47 ± 0.01</td>
</tr>
<tr>
<td>WB40-15</td>
<td>1.51 ± 0.02</td>
</tr>
<tr>
<td>WB40-30</td>
<td>1.65 ± 0.04</td>
</tr>
<tr>
<td>WB40-45</td>
<td>1.73 ± 0.02</td>
</tr>
<tr>
<td>WB50-15</td>
<td>1.88 ± 0.02</td>
</tr>
<tr>
<td>WB50-30</td>
<td>1.94 ± 0.01</td>
</tr>
<tr>
<td>WB50-45</td>
<td>1.96 ± 0.01</td>
</tr>
<tr>
<td>WB60-15</td>
<td>1.98 ± 0.01</td>
</tr>
<tr>
<td>WB60-30</td>
<td>2.15 ± 0.03</td>
</tr>
<tr>
<td>WB60-45</td>
<td>2.36 ± 0.05</td>
</tr>
</tbody>
</table>
Micrograph and EDX Spectrum of Warm Water Treated Al 6061

Fig. 5 shows the micrograph of WB-0 (non-treated) sample with a magnification of 5000X and the EDX spectrum is shown in Fig. 6. Only aluminum (Al) and oxygen (O) peaks were detected in this sample. The O peak designates the native oxide of Al. From Fig. 7, the O peak refers to the new oxide layer i.e. aluminum oxide (Al₂O₃) or it might refer to pseudo-boehmite (AlO(OH)) that already discussed in our previous study [12]. That means the warm water treated substrate surface was covered by Al₂O₃ or AlO(OH) layer. The presence of silicon (Si) reveals that siloxane (SiOSi) layer is also formed and bound at the Al surface formed the metallo-siloxane (Al-O-Si) layer.

Fig. 8 shows the micrographs obtained after immersion in 40, 50 and 60°C for the Al 6061 alloy. At 40°C – 15 min, there is no real sign of a microstructure change, but after 30 min, small white nodules uniformly appear all over the Al surface. After 45 min, the white nodule structure, similar to that observed at 40°C – 30 min escalated and increasing apparently. At 50°C, the white nodules become clearer and the number is growing as the treatment time is increased. A similar trend can be seen for the sample treated at 60°C. The white nodules appear clearer compared to that observed at 40 and 50°C. The white nodules might denote to the Al₂O₃ or AlO(OH) precipitates.
In this study, as the temperature increased, the layer structure would be expected to coarsen. This would explain why the substrate surface created in warm water treatment show a much coarser pattern. The continuous precipitation throughout the Al surface formed at the three different temperatures would also explain why the substrate surface appears to coarsen as it grows and yet still has a uniform distribution.

**Effect of Warm Water Temperature on Weld Bonding Strength**

The effect of warm water treatment on the single lap shear strength of weld-bonded Al 6061 can be observed in Fig. 9. The warm water treatment has given a significant effect on the weld bonding strength of Al 6061. As temperature of warm water is increased, the single lap shear strength has gradually increased. The factors that influenced the strength of weld-bonded Al alloy are mainly the micro-roughness of the substrate surface caused by the AlO(OH) precipitates and the metallo-siloxane (Al-O-Si) layer. The micro-roughness that given by the pseudo-boehmite precipitates created the mechanical interlocking mechanism between the PU adhesive and the Al substrates. Meanwhile, the metallo-siloxane layer played as an anchor, mooring the PU adhesive layer to the Al substrates. There are two mechanisms of reactions that may occur between the adhesive and warm treated Al substrate PU which may contribute to the adhesion. First, through the mechanism of the reaction between PU chains with a metallo-siloxane layer which produced a covalent bond between nitrogen (N) atom of silane layer with the carbon (C) atom from the urethane chain. Jena [13] also believed the same
mechanism between PU-urea coatings with a silane modified zinc oxide (ZnO). The second mechanism involves the reaction between disiocyanate (which has not reacted with the polyol) with a hydroxyl (OH) group of siloxane layer and produced urethane chains that covalently bonded to a silicon (Si) atom. The proposed mechanism of adhesion between the PU adhesive and Al substrate are shown in the Fig.10.

**Figure 9:** Weld bonded lap shear strength of non-treated and warm water treated Al 6061 at various temperatures.

![Graph](image)

**Figure 10:** Proposed mechanism of adhesion between PU adhesive and warm water treated Al 6061.

**Effect of Treatment Time on Weld Bonding Strength**

Fig. 11 displays the significant effect of treatment time on the weld bonding strength. Obviously, the longer treatment time will result in higher weld bonding strength. This is explained by the phenomenon of pseudo-boehmite precipitation on the Al substrate. The longer immersion time has driven more chances for a continuous process of pseudo-boehmite precipitation throughout the Al surface. This is also related to the mechanical interlocking mechanism which can be said as a dominant factor in affecting the weld bonding strength. Petrie [14] claimed that the micro irregularities on the substrate surface provide a barrier to the propagation of crack due to shear load. Thornton et al. [15] suggested that etched AA5754 surfaces revealed a uniform oxide layer that gave the most reliable weld strength and surface resistance. This proposition agreed upon by Pickering and Hart [16] and Li et al. [17].

![Graph](image)

**Figure 11:** Weld bonded lap shear strength of non-treated and warm water treated Al 6061 at various treatment times.

**Figure 12.** Comparison of fractured surfaces of non-treated and warm water treated samples at magnification 50X, (a) WB-0, (b) WB40-15, (c) WB50-30, and (d) WB60-15.

**Fractured Surface Analysis**

Fig. 12 – 14 were taken from the adhesive bonded parts. Fig. 12 shows a comparison micrograph of fractured surfaces of the non-treated sample and part of warm water treated samples at a magnification of 50X. It is clearly seen that the
samples have shown similar fracture mode that is more likely mixed mode fracture. The mixed mode fracture is a combination of adhesive and cohesive failures. It is meaningful to remark that the cohesive failure is unlike the adhesive failure. Cohesive failure is an interlayer failure that occurs inside the adhesive layer whereas the adhesive failure is an interfacial failure happens in the middle of the substrate and adhesive layer. From Fig. 13, it is observed that a mixed mode of failure occurred for sample non-treated sample (WB-0) that includes of adhesive and cohesive failures. This is approved by the EDX result which the fractured surface of WB-0 contains Al element. Meanwhile, the presence of C, N and O elements proved that the PU adhesive still intact on the Al 6061 substrate surface.

Fig. 13: Fractured surfaces of non-treated (WB-0) sample.

Fig.15 shows the fractures at the spot welded failure area. It is observed that at the WB-0 (non-treated) sample exhibits partial interfacial fracture. The fracture occurs when the weld separated through the faying surface. This phenomenon indicates that the current passing during RSW was not easy to penetrate the adhesive layer due to its low electrical conductivity. The warm water treated samples (WB40-30 and WB50-15) exhibit similar failure mode i.e. partial interfacial fracture or more likely no fusion failure mode which means there is no formation of a solid-state bond or a weld nugget during the RSW process. Meanwhile, the WB60-45 shows the result in a full-interfacial fracture. That means the weld nugget experiences a complete fracture through the faying surface. These phenomena indicate that warm water and silane treatment has no significant effect on the spot welded fracture. However, the warm water treatment has affected the weld bonding fracture through the adhesive bonded area. This occurrence has indirectly contributed to the weld bonding strength. Nevertheless, Han et al. [18] have proven that the surface condition of the sheet material produced has a significant effect on the electrode condition, weld quality, and RSW process. This was verified by Al-Naimi et al. [19] in their study on the influence of surface pretreatment in resistance spot welding of aluminum AA1050. They also highlighted that the weldability and electrode lifetime were greatly dependence on the surface condition of the aluminum sheets.

Alas, in spot welds and automotive industry, both partial and full interfacial fractures have been intolerable for many years because this type of fracture has lower tension strength and impact properties of the welds. Furthermore, the weld button can be significantly smaller than the fused area due to the current range reduced by the partial interfacial fractures. Thus it requires higher minimum welding current to achieve the minimum button size. The factors that promote such fractures are predominantly spot weld parameters for instance weld time, weld current, welding pressure and the electrode materials. However, these parameters of spot weld depend on the types of adhesive material used in the weld bonding. Khan et al. [20] in their study on weld bonding of Al 6061 by using synthesized epoxy adhesive concluded that the optimum welding current was 20 kA with an optimum welding time of 8 cycles. Meanwhile, Pereira et al. [21] used commercial epoxy adhesive in their investigation on fatigue strength of the weld-bonded Al joints. The pull out failure mode has successfully achieved by using welding time of 2 – 3 cycles with the welding current 26.4 – 26.9 kA. They also concluded that the fatigue strength of the weld-bonded joints is moderated by adhesive bonding. Therefore, the optimization of weld bonding parameters should consider both adhesive and spot weld parameters to achieve a good weldability, adhesion strength hence the excellent weld bonding can be succeeded.
Figure 14: Fractured surfaces of warm water treated samples at various temperatures, (a) WB40-15, (b) WB40-30, (c) WB40-45, (d) WB50-15, (e) WB50-30, (f) WB40-45, (g) WB60-15, (h) WB60-30, and (i) WB60-45
**CONCLUSION**

Weld bonding strength is found to demonstrate an interesting dependence on warm water treatment followed by silanization in relation to adhesive bonding. However, these treatments did not indicate a significant effect in relation to the quality of spot weld joint. Some modification to the adhesive mixture needs to be taken consideration in further study to solve this problem. The optimization of spot weld parameter is also very important to ensure the quality and integrity of weld bonding.

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43–55.


