The influence of modified annealing during the galvanizing process on the resistance spot welding of the CMn1.8Si advanced high strength steel

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Abstracts

Annealing under an atmosphere with controlled oxygen partial pressure is an effective way for improving reactive wetting during the continuous galvanizing process. Hence, understanding the effects of this annealing step on further manufacturing processes such as welding is essential. The present work has shown that the formation of internal oxides during a controlled atmosphere galvanizing process reduced the resistance and heat input resulted in delaying nugget growth in resistance spot welding, compared to the as-received condition. Furthermore, it was shown that by annealing and subsequent zinc coating, the tensile-shear peak load was decreased under the same welding parameters. The nugget formation mechanism of different surface conditions showed a good correlation with dynamic resistance profiles.

Keywords: Internal oxides; zinc coating; resistance spot welding; surface condition; continuous galvanizing process
1. Introduction

Advanced high strength steels (AHSSs) have developed for automotive applications to meet requirements for safety, efficiency, emissions and quality at relatively low manufacturing and material cost [1]. To maintain the integrity of the vehicle structures, it is required to protect AHSSs from corrosion. The most common processing technique used to protect steel components is continuous galvanizing in which the steel sheets pass through an annealing heat treatment in a controlled, reducing atmosphere before dipping in a molten Zn (Al, Fe) bath [2,3]. However, during the conventional annealing process atmosphere, alloying elements such as Mn, Si, and Al are selectively oxidized in the form of external oxides on the surface sheets [4]. The presence of these surface oxides prior to immersion in the Zn (Al, Fe) molten galvanizing bath compromises reactive wetting resulting in bare spot defects and unacceptable coatings [5,6]. Furthermore, these external oxides may have a detrimental effect on further manufacturing processes such as forming and welding. There are several approaches advocated on reducing the external selective oxidation of the Mn and Si during continuous galvanizing process such as flash coating [7], annealing under NH₃ atmospheres prior to hot-dip galvanizing [8] and modifying the alloying elements [9,10]. Another effective manner for the improvement of the reactive wetting is controlling the dew point in the annealing furnace and therefore the oxygen partial pressure of the furnace resulted in a decrease in the thickness of the external oxides [5,11,12]. Recently, Mousavi et al. [13] showed that with increasing the atmosphere dew point from 50 °C to the +5 °C, internal oxides were formed rather than external oxides that improved the reactive wetting [14].

Resistance spot welding (RSW) is used for 90% of the joints in a typical automobile body-in-white [15]. The principle of the RSW process is based on the generation of heat by applying an electric current to cause melting and nugget formation at the interface of two metal sheets. The electrical resistance of the sheets is the most important factor determines the magnitude of heat and is very sensitive to the surface conditions such as roughness, oxides films and coating [16]. There have been several works characterized the contact resistance of the steel sheets during RSW [17–20]. Saha et al. [21] investigated the effects of Al-Si and zinc coatings on the heat generation and nugget formation of the hot press forming steels. The larger electrode contact diameter and nugget sizes were the main differences between Al-Si and zinc coating samples. Ighodaro et al. [22] compared the mechanical properties of the Al-Si and galvannealed coatings of resistance spot welded hot stamping steel joints. They found that while the coating significantly affected the welding current required for attaining an acceptable nugget size, it did not affect the peak load during the lap-shear test. In another study, Ighodaro et al. [16] compared the effects of two different coating types (Al-Si and galvannealed coating) on the dynamic resistance profiles before and after hot stamping process. It was found that while the galvannealed specimens exhibited higher resistance than Al-Si coated specimens in the as-received condition, the Al-Si specimens exhibited higher resistance after hot stamping. Recently, Han et al. [23] investigated the effects of internal oxidation on the weldability of CMnSi steels. Based on their work it was found that the formation of the molten metal at the contact surface of the internal oxidized sample reduced the contact resistance.

According to the above researches, while the effects of different coatings on the nugget formation and mechanical properties of the AHSSs is well investigated, there is limited work on the effects of the higher-dewpoint annealing atmosphere continuous galvanizing process on the RSW of
AHSSs. Therefore, the objective of the present work is to investigate the effects of this modified annealing during continuous galvanizing process on the nugget formation mechanism, microstructure and mechanical properties of the resistance spot welding of the CMn1.8Si dual phase steel. It was found that the formation of the internal oxides under relatively high oxygen partial pressure (pO_2) atmosphere reduced the resistance as well as heat input compared to the as-received sample. Hence, the nugget size and the maximum tensile-shear load decreased in the annealed and coated samples compared to the as-received condition under the same welding parameters.

2. Experimental details

2.1 Material

The as-received material was cold-rolled CMn1.8Si sheet with 1.0 mm thickness. The chemical composition of the investigated steel is shown in Table 1.

| Table 1. Chemical composition (wt. %) of the as-received steel |
|---------------|--------------|----|---|---|----|
| C  | Mn  | Si  | Al  | Ti  | Fe |
| 0.10 | 1.96 | 1.80 | 0.05 | 0.01 | Bal. |

2.2 annealing and galvanizing process

The selective oxidation process was performed by the McMaster Galvanizing Simulator (MGS, Iwatani) in a N_2-5vol.%H_2 process atmosphere using a dew point of -5 °C (see table 2). This process comprised heating the samples to 773 K (500 °C) at 15 K/s, followed by heating to the intercritical annealing temperature (IAT) at 5 K/s and holding at this temperature for 120 s. The samples then were cooled to 738 K (465 °C) at -20 K/s, holding at 738 K (465 °C) for 20 s and finally cooled to room temperature by N_2 jet cooling. The process for galvanizing samples was the same to the selective oxidation, however, another step was added before the final cooling, which comprised dipping the samples for 4 s in an Fe saturated 0.2 wt.% dissolved Al zinc bath at 733 K (460 °C). More details about selective oxidation and galvanizing processes can be found elsewhere [6,24]. The samples are coded as the AR, IO and IO+C according to their surface conditions of as-received, annealed prior to galvanizing, annealed, and coated, respectively.

| Table 2. Annealing process parameters |
|-----------------------------|------------|-----------|----------|
| IAT (°C)       | Dew point (°C) | pH2O/pH2  | PO2 (atm) |
| 837            | -5          | 0.083     | 3.91×10^{-21} |

2.3. Resistance spot welding

Resistance spot welding was performed using a medium-frequency direct current (MFDC) welder with Bosch control timer with Cu-Cr dome radius type electrode (AWS D8.9 standard) with 6 mm
tip diameter under a constant water cooling rate of 6 L min\(^{-1}\). Welding parameters were selected according to the AWS D8.9 standard (see Table 3) [25]. Before welding, the samples were cleaned with ethanol to remove surface contaminations. Dynamic resistance data between electrodes and samples during welding were obtained using instantaneous voltage and current measured using the Bosch controller.

Table 3. Weld parameters in accordance with AWS D8.9

<table>
<thead>
<tr>
<th>Force (kN)</th>
<th>Welding time (ms)</th>
<th>Holding time (ms)</th>
<th>Current (kA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>250</td>
<td>167</td>
<td>8</td>
</tr>
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2.4. Characterization

Samples for microstructural observation were prepared through conventional procedures, i.e. mounting in conductive epoxy resin (Polyfast), grinding with SiC sandpaper up to 1200 grit, polishing to 1 µm diamond spray and etching with 2% Nital (2% HNO\(_3\) + 98% ethanol). Microstructures were characterized using optical microscopy (OM) and field emission scanning electron microscope (FESEM, UltraZeiss) techniques. The working distance and accelerating voltage of the FESEM were about 9.8–10.8 mm and 20 kV, respectively. To investigate the effects of surface condition on the nugget formation, welding process was interrupted at the different welding times followed by separation of the welded samples by a chisel. Then, the faying surfaces (that comprised the interface between the two sides of the RSW joint) of the samples were observed by stereomicroscope. Vickers microhardness test was conducted using a Clemex CMT automated hardness tester according to ASTM E384. Microhardness measurements were captured by applying a load of 200 g for 15 s. Tensile lap-shear testing with the sample dimension of 60 mm×20 mm (length × width) was carried out using an Instron tensile tester at a crosshead speed of 1mm/min.

3. Results and discussion

3.1. Surface characteristics

The surface characteristics of the CMn1.8Si steel are shown in Fig. 1. Two types of oxide networks existed in the CMn1.8Si steel after the annealing under controlled atmosphere process. The first oxide type is the continuous oxides formed along the grain boundaries (intergranular oxides) and the second type is the transgranular oxides formed into the grains with spherical morphology (Fig.1 (b)). It was reported that both intergranular and transgranular oxides comprised a multi-layer configuration, where the SiO\(_2\) core surrounded by MnSiO\(_3\) outer shell [12]. Fig. 1 (c) shows the SEM micrograph together with energy-dispersive X-ray spectroscopy (EDS) analysis of the as-annealed samples after galvanizing process. As can be seen, there is no significant changes in oxides layer thicknesses of pre-immersion and post-immersion conditions. The average of thickness of the internal oxides and coating layer were 3.5±0.4 µm and 9.1 ± 0.7 µm, respectively.
Fig. 1. SEM micrographs of the surface characteristics of the CMn1.8Si steel, (a) as-received, (b) annealed prior to zinc immersion and (c) annealed sample followed by zinc immersion and EDS measurement across indicated interface.
3.2. Effects of surface conditions on the nugget size

Fig. 2 shows the effects of surface conditions on the nugget size and dynamic resistance profiles of the investigated steels. As shown, the AR and IO+C samples possess the highest and the lowest nugget size under the same welding parameters. This difference in the nugget size is attributed to the differences in resistance and heat input during welding. As shown in Fig. 2, the dynamic resistance profiles consist of two main regions as region A with the surface condition dominated mechanism and region B with the nugget growth dominated mechanism. As illustrated in Fig. 2, a significant difference in dynamic resistance can be observed in the region A under the same welding parameters. The resistance rapidly increases to the maximum value (β point in Fig. 2) as competitive results of surface film breakdown, asperity softening and bulk temperature increasing in all samples [18]. However, the AR sample possesses the highest β point compared to IO surface condition. By annealing under controlled atmosphere, the external oxides transmitted to the internal oxide networks resulted in lower resistance of the IO sample compared to the AR condition. Furthermore, the lowest resistance in the IO+C sample is attributed to the presence of the zinc coating on the faying surface as well as internal oxides which strongly reduced the contact resistance [9]. Moreover, the position of the β point is shifted to the longer welding time in the IO and IO+C samples resulted in delaying in nugget growth. Based on the dynamic profiles, it is clear that the nugget started to form at the shorter welding time in the AR condition with comparison to those IO and IO+C samples. Therefore, the AR condition had more time for nugget growth resulted in the larger nugget size compared to other samples. Fig. 3 shows the variation of the $R_\beta$ (resistance value in the β point) as well as resistance drop (difference of the peak resistance with the resistance value at the end of welding, see Fig. 3) with versus nugget size for the investigated steels. As shown, with increasing $R_\beta$ and resistance drop values, the nugget size is increased. In addition, the IO+C possesses the lowest value of resistance drop resulted in the smallest nugget size.
Fig. 2. The variation of the nugget size and dynamic resistance profiles of the investigated steels after RSW process; (AR: as-received, IO: annealed prior to galvanizing and IO+C: annealed, and coated).

Fig. 3. Variation of the (a) maximum resistance ($R_\beta$) and (b) resistance drop versus nugget size in different surface conditions of the investigated steels; (AR: as-received, IO: annealed prior to galvanizing and IO+C: annealed, and coated).
3.3. Effect of surface conditions on the nugget formation

To clarify the effects of surface conditions on the nugget formation mechanism, the welding process was interrupted at different welding times to observe the weld development (Fig. 4). From Fig. 4 it is observed that the nugget was not formed at welding times up to the 30 ms in all surface conditions. However, at the 40 ms, a small area of the nugget (highlighted with white line in Fig. 4) was formed in the AR sample. The presence of the molten nugget in the AR sample showed a good correlation with the dynamic resistance profiles (Fig. 2). On the other hand, after 10 ms, the coating melting was not observed in the faying surface of the IO+C sample. However, after 20 ms, the coating at the faying interface begun to melt. In addition, electrode pressure squeezes the molten zinc away from the center. This molten zinc provides a liquid contact which further reduce the reduction. After 30 ms, the zinc was completely melted at the center and the steel sheet faying interface appeared. Finally, at the 40 ms, the HAZ area of the steel sheet surrounded by molten zinc ring grown and the melting at the flying surface of the steel sheets was not observed in this sample.

Fig. 4. Macro photographs of the initial melting at the faying surfaces in different welding times; (AR: as-received, IO: annealed prior to galvanizing and IO+C: annealed, and coated).
Fig. 5 shows the micrographs of the cross-section area of the samples after 40 ms, 50 ms, and 60 ms. As shown, there is no observed weld nugget in the IO+C samples up to 60 ms welding time. As mentioned, the first molten nugget formed at the area near to the β point. Therefore, according to the dynamic resistance profiles (Fig. 2), the β point of the IO+C samples occurred around 100 ms and consequently, there is no melting in this surface condition before this time. However, with increasing welding time, the HAZ area (highlighted in white lines in Fig. 5) is increased in this sample which is attributed to applying more heating in longer welding time. On the other hand, with increasing welding time up to 50 ms, the nugget (highlighted in yellow dashed line in Fig. 5) was formed in the AR and IO samples, however, the actual joint happened only in a small area in the IO sample. Further increasing welding time up to 60 ms caused the nugget growth and increasing of the nugget size. Fig. 6 shows the OM and SEM micrographs of the interfacial area of the IO sample near the HAZ region in different welding times. It can be seen that the bonding layer (corona bond in Fig. 6) is observed in the IO sample started from the outer periphery of the fusion zone and propagated to the HAZ region in all welding times. In should be mentioned that these corona interfaces did not observe in the AR sample. The formation mechanism of the corona bonds is attributed to bonding through plastic deformation of the faying surfaces [26]. In the RSW process, plastic deformation resulting from the interaction between the electrode force and thermally-softened solid metal at peripheral area of the fusion zone provides corona bonds which resists against sheet separation. The surface condition significantly influences pressure contact and subsequently formation of the corona bond [27]. It was reported that the formation of the corona bond is significantly influenced by oxides layer thickness in which with increasing surface oxide film thickness the bonding ability was decreased [28]. Since the IO sample exhibits lower external oxides due to the controlled atmosphere annealing, the corona bond was observed in this sample. Fig. 6 shows the detailed micrographs of the corona bonds after different welding times. As illustrated in Fig. 6 (a), the two sheets interface partially melted to each other. However, in regions where melting did not occur, oxide coarsening indicated that the applied heat was insufficient to form partially joined regions. In addition, it can be seen that the intergranular morphology of the internal oxides changed to an evenly distributed spherical shape due to oxides coarsening (Figure 6 (c)).
Fig. 5. Optical micrographs of the nugget cross sections welded at the 10 ms to 40 ms in three different surface conditions; (AR: as-received, IO: annealed prior to galvanizing and IO+C: annealed, and coated).

Fig. 6. OM and SEM micrographs of the interfacial area of the IO sample showing the formation of corona bond after (a) 40 ms, (b) 50 ms and (c) 60 ms.
3.4. Microstructural analysis

Fig. 7 shows the microstructures of the AR and IO samples after 250 ms welding time. As shown, the corona bond is observed only in IO sample. It should be noted that the only difference between the fabrication parameters of the IO and IO+C samples was dipping the IO+C samples for 4 s in zinc bath at 733 K (460 °C). Therefore, it was observed that the detailed microstructures of the IO and IO+C samples were similar because there was no effect of the low temperature (465 °C) and short (4 s) galvanizing process on microstructure. On the other hand, the microstructure of the base metal (BM) in the AR condition (Fig. 7b) is comprised of ferrites and pearlites elongated in the rolling direction. However, according to the intercritical annealing, the BM microstructure of the IO sample (Fig. 7e) consists of the ferrite matrix with the chain-network martensite. The microstructures of the intercritical heat affected zone (ICHAZ) in the AR (Fig. 7c) and IO (Fig. 7e) samples consist of the ferrite and martensite islands. However, the martensite morphology of ICHAZ region in the IO sample is different compared to the AR sample. It was reported that the initial microstructure has a significant influence on the morphological distribution of the two phases. DP steels formed from the initial cold-rolled ferrite-pearlite structure consisted of recrystallized ferrite surrounded by martensite network [29]. In contrast, the microstructure developed from the initial duplex ferrite-martensite structure consists of ferrite matrix and chain-networks martensite. Furthermore, according to the higher temperature in the other regions such as upper critical HAZ (UCHAZ) and fusion zone (FZ), the microstructures of these regions were similar in all surface conditions.
3.5 Mechanical properties and failure analysis

Microhardness profiles of the investigated steels are shown in Fig. 8 (a). The hardness measurements showed that AR and IO+C conditions possess the highest and the lowest hardness in the BM region, respectively. While it is expected that the ferrite/martensite structures had higher hardness values, however, due to the large ferrite grains (7.9 ±1.2 µm) with small size martensite networks (2.4 ±0.3 µm) the overall microhardness of the IO condition is smaller than the AR
sample. It should be noted that the short annealing during zinc immersion caused a reduction in hardness values in the IO+C samples in the BM region. The hardness reached to its maximum values (410-430 HV) in the FZ where a fully martensite structure is present. Furthermore, a sharp reduction in the hardness is observed in the HAZ region compared to the FZ in all investigated steels, however, the HAZ hardness is still higher than those BM regions. The reason for this behavior can be traced to the microstructural features of the BM and HAZ regions developed during the RSW process. As illustrated, the BM structures composed of the elongated ferrite and pearlite (in the AR condition) and coarse ferrite with low martensite volume fraction (in the IO samples). In contrast, the microstructure of the HAZ regions consisted of fine-grained ferrite surrounded by high martensite volume fraction (approximately 60 percent). Such the ferrite-martensite duplex structures with smaller ferrite grain size and higher martensite volume fraction provided the higher hardness compared to the BM microstructures.

Fig. 8 (b) shows the variation of the maximum tensile-shear load versus nugget size. As shown, the AR and IO+C samples possess the highest and the lowest maximum load, respectively. It is well established that the nugget size is the most important controlling factor determining the mechanical strength of the welds [30]. However, the failure mode significantly affects the peak loads during the lap-shear test. Since all investigated steels failed with the pullout failure mechanism, it seems that the dominant mechanism affected the mechanical properties of the investigated steels was the nugget size. It is clear that with increasing nugget size the peak load is increased in the linear behavior. Fig. 8 (c-e) reveals the representative SEM micrographs of the fracture surface of the lap-shear specimens which showed typical ductile dimple morphology. As shown in Fig. 8e, the AR sample possess a finer dimple size compared to those IO and IO+C samples. The number of dimples per unit area on the fracture surfaces depends on the number of nucleation sites. The finer the grain size, the more sites for void nucleation. The finer ferrite grains size and martensite islands in the ICHAZ region of the AR sample caused higher density of nucleation sites and finer dimple size.
4. Conclusion

This work highlights the influence of annealing under controlled atmosphere during continues galvanizing process on the resistance spot welding of the CMn1.8Si advanced high strength steel. Annealing and subsequently coating under controlled pressure atmosphere reduced the maximum resistance value ($\beta$ point) and shifted it to the longer welding time compared to the as-received sample. Hence, the nugget size and the maximum tensile-shear load were decreased under the same welding parameters. This suggests that by annealing under relative high oxygen partial pressure in the continues galvanizing process, higher welding current or longer welding time is required to compensate the reduction of heat generation caused by the surface changes.
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