New Coating Development for Press Hardening Steels

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INTRODUCTION

The process of hot stamping for press hardening steels involves heating the blanks to elevated temperatures for austenitization in a furnace, followed by transferring the blanks to a stamping press for simultaneous die forming and quenching operations [1-2]. Typically, the press hardening boron steels utilized for hot stamping of structural parts are hot dip coated with AlSi10Fe3 alloy (aluminized), aimed at avoiding the formation of scale during heating in the furnace.

Studies [3-4] have shown that during heating in the furnace, the AlSi10Fe3 coating melts at a temperature of approximately 575°C. This phenomenon facilitates the outward diffusion of Fe and Mn from the steel substrate into the molten AlSi10Fe3 layer, leading to the formation of intermetallic phases with higher melting temperatures. Research [5-7] has demonstrated, through the use of an interrupted heating technique at various temperatures and soaking times, that the inward diffusion of Al and Si into the steel substrate is triggered above the austenite starting temperature (AC1). The coating development progresses above the austenite finish temperature (AC3), where the outward diffusion of Fe and Mn from the steel substrate into the AlSi10Fe3 coating results in the formation of Al-Fe-Si intermetallic phases at the coating surface, and the inward diffusion of Al and Si from the coating into the steel substrate causes the growth of the interdiffusion layer at the steel-coating interface. Longer soaking times above AC3 temperatures result in reduced performance during welding and painting operations of the hot formed parts, leading to an increase in the thickness of the interdiffusion layer and the total coating thickness. Industry standards, therefore, restrict the coating development during the residence time in the furnace to a maximum interdiffusion layer thickness of 12 to 16 microns and a maximum total coating thickness of 30 to 50 microns [8-9]. These coating development attributes are directly related to the performance in welding and painting operations and the corrosion resistance of hot stamped parts.

In this study, a new coating for press hardening steel (PHS) is presented as an alternative to the traditional aluminized AlSi10Fe3 coating used in the hot stamping process. The new coating is a heat-resistant paint that consists of a pre-ceramic polymer and aluminum powder as an active filler [10-13]. The heat-resistant paint coating was applied to a 22MnB5 steel substrate with a thickness of 0.9mm in coil form using an industrial roller coating pre-paint line. A 22MnB5 PHS with a thickness of 0.9mm and coated with a typical hot dip aluminized coating was used as a baseline for comparison purposes. The behavior and development of the new coating during heating in the furnace were analyzed using differential scanning calorimetry (DSC) and Continuum Depth Profile with Glow Digital Spectrometry (CDP-GDS). The surface phase composition of the new coating before and after heating for the hot stamping process was characterized using scanning electron microscopy, and energy-dispersive X-Ray spectroscopy (SEM-EDX). Results from the welding, E-coating, and corrosion resistance studies performed on the hot stamped parts are presented in this paper, along with a discussion of the feasibility of a window process for the residence time in the furnace for the new PHS coating.
EXPERIMENTAL WORK

Industrial Hot Stamping Trial
Blanks for a current production part were cut using a laser cutting process from both the roller paint coated coil and the production hot-dip aluminized coated coil. These blanks were then subjected to an industrial hot stamping trial that was carried out using an AP&T hot stamping production cell, which included a linear transfer system for loading and unloading the blanks, a multi-layered furnace, and a hydraulic press. The aluminized coated blanks underwent an optimized thermal cycle with a production intent, consisting of a 3-minute furnace residence time at 930°C. The paint coated blanks were loaded into the furnace at a soaking temperature of 930°C and subjected to furnace residence times of 2 minutes and 5 minutes for this study. After the furnace residence time, the blanks were unloaded and transferred to the press within 10 seconds, followed by die forming and quenching with a 3-second die dwell time.

K-type thermocouples were welded onto the paint and aluminized coated blanks and a data acquisition system was used to determine the temperature profiles of the 22MnB5 steel substrate during the furnace residence time. The experimental results are plotted in Figure 1A, which show the temperature profiles of the paint and aluminized coated blanks.

![Figure 1A]()

Figure 1, A) Blank temperature profiles during heating in the furnace of the paint and aluminized coated 22MnB5 steels; B) hot stamped parts, B1- aluminized coating, B2- paint coated 2 minutes furnace residence time and B3- paint coated 5 minutes furnace residence time

It is observed from Figure 1A that the paint coated blank reached the furnace soaking temperature of 930°C in approximately 25 seconds, with an average heating rate of 36°C/s. On the other hand, the hot-dip aluminized coated blank required approximately 130 seconds to reach the furnace soaking temperature, resulting in an average heating rate of 7°C/s. These results can be attributed to differences in the radiative properties of the two coatings, such as surface emissivity and absorptivity, as well as the melting and solidification of the aluminized coating during heating in the furnace. These findings are in good agreement with the discrepancies observed between the DSC signals for the paint and aluminized coated samples, confirming that the paint coating does not melt during the furnace residence time.

Figure 1B presents the surface appearance of the hot stamped parts of the paint coated 22MnB5 blanks subjected to 2 minutes and 5 minutes of furnace residence time, as well as the hot-dip aluminized coated 22MnB5 blank after 3 minutes of furnace residence time. The results indicate that the paint coating demonstrates good thermal stability at high temperatures and good coating adhesion during hot plastic deformation, as no visual surface defects such as paint exfoliation, blisters, or bare spots were observed on the surface of the hot stamped parts.

Industrial Relevance - Weldability study
A study was conducted to evaluate the weldability of paint coated 22MnB5 PHS, using 600 coupons with dimensions of 50mmx150mm extracted from hot-formed parts produced with a five-minute furnace residence time. The study was using Ford Laboratory Testing Method FLTM BA113-08 as a reference and the main scope was to provide industry reference guidelines for resistance spot welding (RSW) of paint coated hot formed coupons in a 2T homogeneous stack up configuration.

For a material thickness of 0.9mm, the minimum required weld size diameter was determined to be 3.7mm, using 16x20mm ISO electrodes. The weld current range methodology adopted from the FLTM BA113-08 consisted in determining the
minimum current intensity \((I_{\text{min}})\) for which the minimum weld size diameter \((D_{\text{min}})\) of 3.7mm is obtained, and the maximum current intensity \((I_{\text{max}})\) before expulsion occurs that produces the maximum weld size diameter \((D_{\text{max}})\).

The results of the weldability study, shown in Figure 2, demonstrate that a 2.0kA current range can be obtained for paint coated hot formed coupons in a 2T homogeneous stack up configuration, using a welding current waveform with two pre-pulses of 5ms and 200ms at 2.5kA.

An example of the welding current waveform is presented in Figure 2A. Figure 2B presents the current range obtained for the 2T homogeneous stack up configuration using an 8mm diameter electrode-face and 400ms welding time. It was observed that the minimum weld size diameter \((D_{\text{min}})\) of 3.8mm was obtained at a minimum current \((I_{\text{min}})\) of 3.3kA, while the maximum weld size diameter \((D_{\text{max}})\) of 6.1mm was obtained at a maximum current \((I_{\text{max}})\) of 5.3kA resulting in a 2.0kA current range.

To evaluate the mechanical properties of the welds, Shear Tension and Cross Tension tests were conducted on specimens produced using a welding schedule developed for the minimum weld size diameter \((\text{MWS})\). The average peak load obtained from the Shear Tension tests was 7.15KN, while the average peak load obtained from the Cross Tension tests was 1.61KN.

It is worth noting that previous weldability studies on the baseline aluminized coated 22MnB5 PHS with a thickness of 0.9mm, tested according to FLTM BA113-08 requirements, reported a current range of 1.2kA to 1.4kA. These findings highlight the importance of careful selection of welding parameters for optimizing the weld properties and achieving desired results.

**Industrial Relevance - E-coating and Corrosion Resistance Study**

The E-coating and corrosion resistance properties of paint coated 22MnB5 hot formed parts were studied through an evaluation of parts produced with 2 minutes and 5 minutes furnace residence time, without undergoing shot blasting after the hot stamping process. The hot stamped parts were subjected to a current production E-coating process.

Figure 3, A) Paint coated 22MnB5 hot stamped part carried by a BIW through the E-coating production line, B) after E-coating process, B1 - 2 minutes respective B2 – 5 minutes furnace residence time
The paint coated 22MnB5 hot formed parts were securely attached onto the floor of a body in white (BIW) and carried through the E-coating production line, as seen in Figure 6A. A visual examination of the E-coated parts was conducted, and no surface defects were observed. An image of the E-coated hot stamped parts produced with 2 minutes and 5 minutes of furnace residence time is shown in Figure 3B.

The corrosion resistance performance of the E-coated hot stamped parts with 2 minutes and 5 minutes of furnace residence time was evaluated based on the L-467 Scribe Creep Test and FLTM BI 169-01 specifications. The evaluation process involved cutting six panels from each part and scribing a diagonal line on each panel. The panels were then subjected to an accelerated cyclic corrosion testing for up to twelve weeks. The scribe creep was measured at six and twelve weeks. Figure 4 illustrates the scribed panels before and after six weeks of accelerated cyclic corrosion testing.

![Figure 4, Example of the scribed panels: A) before and B) after 6 weeks of accelerated cyclic corrosion testing, panels sampled from hot formed part produced with 2 minutes furnace residence time](image)

The L-467 Scribe Creep Testing specification for low alloyed steels and iron castings requires that the average of the scribe creep measured at 6 weeks to be less than 10mm. The results for the L-467 Scribe Creep Testing after 6 and 12 weeks of accelerated cyclic corrosion testing of the panels sampled from the hot formed parts produced with 2 minutes and 5 minutes furnace residence time are summarized in Figure 5.

The data, presented in Figure 5, indicates that the average scribe creep value measured after 6 weeks, respective 12 weeks of accelerated cyclic corrosion increases with an increase in the furnace residence time, and in case of the hot stamped parts produced with 5 minutes furnace residence time, the scribe creep measurements after 6 weeks are marginal to the 10mm limit requirement specification per L-467 Scribe Creep Test.

![Figure 5, Scribe creep measurements after 6 and 12 weeks of accelerated cyclic corrosion testing](image)

Previous studies on the hot dip aluminized 22MnB5 hot stamped production parts have reported a lower average scribe creep measurement of approximately 3.5mm compared to the paint coated samples with a 2-minute furnace residence time. This
Differential Scanning Calorimetry (DSC) Investigations
In the present study, the behavior of the pre-ceramic polymer and aluminum powder coated press hardening steel (PHS) during heating in the furnace was analyzed using Differential Scanning Calorimetry (DSC). DSC is a widely utilized analytical technique that measures the heat flow and temperature changes associated with physical and chemical transitions in materials. The measurement provides both qualitative and quantitative information regarding the exothermic or endothermic processes that occur in the sample.

In a DSC analysis, an endothermic process in the sample leads to a peak in the DSC signal due to the reduction in the sample temperature relative to the reference air temperature. Conversely, an exothermic process releases heat, leading to an inverse peak in the DSC signal due to the increase in the sample temperature compared to the reference air temperature.

In this study, DSC investigations were conducted on heat resistant paint coated and aluminized 22MnB5 steel samples at a constant heating rate of 0.08°C/s. The results of the DSC investigations are presented in Figure 6.

**Figure 6A** shows the thermal reactions that occur in the paint and aluminized coated 22MnB5 steel samples as a function of temperature. **Figure 6B** shows the changes of the normalized mass for the paint and aluminized coated 22MnB5 steel samples as a function of temperature.

The results from the DSC investigation on the AlSi10Fe3 (aluminized) coated 22MnB5 steel sample are presented in Figure 6A. The DSC signal of the aluminized coated sample exhibits a peak starting at around 580°C and ending at approximately 640°C, which corresponds to the melting point and solidification of the AlSi10Fe3 eutectic coating. A second DSC signal peak is observed, starting at around 720°C and ending at approximately 840°C, which corresponds to the phase transformation of ferrite to austenite in the 22MnB5 steel substrate.

The DSC signal of the paint coated 22MnB5 sample shown in Figure 6A, exhibits a smooth inverted peak between 485°C and 530°C, corresponding to a low energy exothermic reaction in the paint coating. The second peak, starting at around 720°C and ending at approximately 840°C, is the same as the aluminized coated sample and corresponds to the phase transformation of ferrite to austenite in the 22MnB5 steel substrate. The absence of the melting point of the paint coating during heating is reflected in the differences between the DSC signals for the paint and aluminized coated samples.

The normalized mass increase of the paint and aluminized coated 22MnB5 steel samples as a function of temperature is shown in Figure 6B. The mass increase during heating is due to the adsorption of oxygen from the furnace atmosphere and to the formation of surface oxides. The results show that at temperatures above 550°C, the oxygen adsorption of the paint coated sample is higher than that of the aluminized coated sample. This indicates that the paint coated 22MnB5 steel will experience
higher surface oxidation and oxidation throughout the coating thickness during heating in the furnace for the hot stamping process.

**Glow Digital Spectrometry (GDS) Analyses**

The development of the paint coating during heating was analyzed using a LECO Glow Discharge Spectrometer (GDS 950). The newly developed GDS 950 has the capability of determining the chemical composition of 40 elements using a combined Radio Frequency and Direct Current Glow Discharge (RF/DC) lamp. A Continuum Depth Profile (CDP) method was developed to analyze the heat resistance paint coating, using the RF glow discharge and elemental sputtering rates. The method provides both qualitative and quantitative analysis of the elemental chemical composition throughout the coating thickness and into the steel substrate.

The coating development during the furnace residence time was investigated using the CDP method and the study was conducted on coupons extracted from the hot-formed parts. Figure 7 presents the elemental profiles of the main chemical elements C, O, Al, Si, Fe, and Mn, obtained by sputtering through the coating thickness and into the steel substrate, under the sample conditions of 0 minutes, 2 minutes, and 5 minutes furnace residence time.

![Figure 7, Elemental profiles of the paint coated 22MnB5 PHS for the: A) 0 minutes; B) 2 minutes; C) 5 minutes furnace residence time conditions](image)

The elemental profiles of both oxygen and iron as depicted in Figure 7 suggest that during the furnace residence time, the oxygen from the furnace atmosphere diffuses into the coating, while iron from the steel substrate migrates outward into the coating. The iron concentration, measured at the proximity of the coating surface, demonstrates an outward diffusion trend, increasing from an initial value of 2%Fe for 0 minutes furnace residence time to 6%Fe and 20%Fe for 2 minutes and 5 minutes of furnace residence time, respectively. A general increase in iron diffusion into the coating is observed as the furnace residence time increases, with the iron elemental profile exhibiting a constant level of approximately 55%Fe at a depth of approximately 0.5 microns from the coating surface. Meanwhile, the oxygen concentration measured near the surface displays an increase from 7%O for 0 minutes furnace residence time to 60%O and 68%O for 5 minutes and 2 minutes of furnace residence time, respectively.

The carbon elemental profile of the paint coating undergoes significant changes upon heating at 930°C, as indicated by the results obtained from Figure 7. The near-surface carbon percentage, which is measured to be approximately 22% for the 0-minute condition, decreases to 3% and 1% for the 2-minute and 5-minute furnace residence time conditions, respectively. Further, the carbon elemental profiles for the 2-minute and 5-minute conditions overlap after a depth of 1.5 microns into the coating. These findings suggest that upon heating at 930°C, the pre-ceramic polymer undergoes to a polymer-to-ceramic conversion.

The results from Figure 7 also show a decrease of silicon concentration with an increase in the furnace residence time. The silicon concentration measured approximately 50% for the 0-minute condition and decreases to 25% and 10% for the 2-minute and 5-minute conditions, respectively. A similar trend is observed for the aluminum elemental profile, which decreases from approximately 23% for the 0-minute condition to 13% and 8% for the 2-minute and 5-minute conditions, respectively, when measured approximately at 1 micron from the coating surface.

The CDP-GDS methods can be utilized to determine the thickness of the paint coating on the steel substrate. The nominal thickness of the paint coating is determined by the intersection of the aluminum and chromium elemental profiles, referred to...
as the Al-Cr intersect. Based on this method, the paint coating thickness of the 0-minute sample was measured to be 7.6 microns, while the thickness of the 2-minute and 5-minute sample conditions were measured to be 7.7 microns and 7.9 microns, respectively. The small differences in the coating thickness measurements between the 0-minute condition and the 2-minute and 5-minute furnace residence time conditions indicates that the formation of an interdiffusion layer between the coating and steel substrate does not occur during furnace residence time.

Scanning Electron Microscopy (SEM)
The near-surface development of the paint coating during the furnace residence time was studied using scanning electron microscopy coupled with energy dispersive spectrometry (SEM-EDS). Phase mapping analyses were conducted on the as-received sample, as well as on samples extracted from the hot-formed parts produced with 2-minute and 5-minute furnace residence times. The SEM-EDS method provides qualitative and semi-quantitative information on the phases present at the near surface of the sample. Figure 8 presents the electron image and associated phase mapping images, along with the semi-quantitative phase volume fractions determined for the 0-minute, 2-minute, and 5-minute furnace residence time samples.

The phase composition of the paint coating at the surface of the 0-minute furnace residence time sample was found to consist of a matrix of silicon-aluminum oxides (approximately 58% SiAlO), islands of silicon oxides (approximately 28% SiCO), and globular aluminum-silicon oxides (approximately 5% AlSiO), crosslinked with a pre-ceramic polymer.

Figure 8, Electron image at 300x magnification, phase mapping and identification and phase volume fractions for the A) 0 minutes, B) 2 minutes and C) 5 minutes furnace residence time samples

The results of the phase mapping analysis show that after a 2-minute furnace residence time at 930°C, the surface of the paint coating is composed of a continuous network of iron-silicon oxides (approximately 19% FeSiO) surrounding a matrix of silicon-aluminum oxides (approximately 60% SiAlO) and globular particles of aluminum-silicon-iron oxides (approximately 4% AlSiFeO) uniformly distributed throughout the network. With an increased furnace residence time of 5 minutes, the paint coating surface further evolves to consist of a matrix of silicon-aluminum oxides (approximately 34% SiAlO) with a network of iron oxide (approximately 17% FeO) and globular iron-silicon oxide (approximately 18% FeSiO), and islands of aluminum-silicon-iron oxides (approximately 9% AlSiFeO) uniformly distributed throughout the network.
The changes of the stoichiometry on the surface of the paint coating during the duration of exposure in the furnace can be attributed to both polymer cross-linking and pyrolysis as well as the diffusion of oxygen from the furnace atmosphere into the coating and iron diffusion from the steel substrate through the paint. SEM-EDS phase mapping analysis further supports these observations by revealing a transformation of the microstructural phases from an amorphous state to crystalline phases. These results, in conjunction with findings obtained from RF-CDP-GDS and DSC analyses, provide insight into the primary mechanisms involved in the development of the paint coating during its residence time in the furnace.

**Paint coating development mechanism**

The as-received paint coating for PHS comprises of a pre-ceramic polymer and aluminum powder as an active filler. Upon heating to 930°C, the polymer undergoes to a pyrolysis process producing an exothermic reaction. During the residence time in the furnace, the coating is subjected to inward diffusion of oxygen from the furnace atmosphere and outward diffusion of iron from the steel substrate, leading to altered phase compositions and morphologies.

A two-minute residence time at 930°C results in an enrichment of Si oxide islands with Fe, leading to the formation of a network of Fe-Si oxides and islands of Al-Si-Fe oxides in a Si-Al oxide matrix. Further increasing the residence time to five minutes results in additional diffusion of Fe from the steel substrate, leading to a modification of the Fe-Si oxide phase morphology from a network to a globular structure, and the formation of a new Fe oxide network. This also leads to an increase in the volume fraction of Al-Si-Fe oxide islands at the expense of the Si-Al oxide matrix.

A comprehensive understanding of the coating development during the furnace residence time is crucial in formulating and refining the paint and roller paint technology, as well as optimizing the hot stamping process for improved weldability and corrosion performance.

**Optimized Hot Stamping Process**

Press-hardening of aluminized 22MnB5 steel is a widely used technique for the production of body-in-white (BIW) structural components. The time-temperature optimization process for this process is limited by the need to produce a fully developed aluminized coating at the end of the heating operation, while also fulfilling the requirements of mechanical properties and weldability.

The use of paint-coated 22MnB5 steel for press-hardening opens up the possibility of optimizing the time-temperature process for complete austenitization of the steel substrate, while also meeting the weldability, paintability, and corrosion resistance requirements. This implies to identify a time-temperature window process, in which the mechanical properties are not affected, and the paint coating development provides improved performance in weldability, paintability and corrosion resistance.

The complete austenitization process of 22MnB5 steel is well documented in literature [14-16] and involves heating the steel above the austenite finish temperature (AC3) to transform the ferrite and perlite to austenite, followed by a soaking time for the dissolution of secondary phases into austenite and the chemical homogenization and grain refinement of austenite.

The differential scanning calorimetry (DSC) results of the current study showed that the mass growth due to oxidation in the paint-coated 22MnB5 specimen increases exponentially with increasing temperature above 830°C. This suggests that furnace soaking temperatures lower than 930°C applied to the paint-coated 22MnB5 specimen would reduce the oxidation process and improve the weldability and corrosion resistance performance.

Preliminary experiments were carried out using a tension-deformation dilatometer (DIL805A/D) [17]. A temperature range of 820°C to 880°C was investigated in 20°C increments, with a heating rate of 30°C/s and a soaking time of 90 seconds. The mini-tensile specimens were then cooled to 650°C in about 10 seconds, subjected to non-isothermal plastic deformation between 650°C and 635°C with 10% plastic strain applied in about 0.5 seconds, and finally quenched to 80°C with a cooling rate of 123°C/s and slow cooled to 30°C in 25 seconds. The mini-tensile specimens were evaluated for their final mechanical properties and microhardness, with three replicates for each condition tested and the average yield strength, tensile strength, and microhardness values reported in Table 1

<table>
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<tr>
<th>Temperature</th>
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<th>840°C</th>
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<th>880°C</th>
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Preliminary results indicated that a 90-second soaking time at a temperature range between 860°C and 880°C, which corresponds to an approximately temperature range of AC3+ (20°C to 40°C), has minimal or no impact on the mechanical properties. The mechanical properties were within the Ford performance specifications for hot-stamped parts.
CONCLUSIONS

A new heat-resistant paint coating for press-hardened 22MnB5 steel (PHS) was investigated as an alternative to the traditional AlSi10Fe3 aluminized coating. The study involved evaluating the new coating from both manufacturing and performance perspectives.

The weldability and corrosion resistance performance of the new coating depend on the furnace residence time. Adequate RSW results were obtained using a waveform welding current with two pre-pulses of 5ms and 200ms at 2.5kA, respectively. The corrosion resistance performance of the E-coated hot stamped parts, evaluated using the L-467 Scribe Creep test, suggested that the paint coated 22MnB5 PHS could be used for interior structural parts located in dry areas if the hot stamped parts are subjected to an optimized furnace residence time and temperature.

The results of DSC, CDP-GDS and SEM-EDS analyses revealed that during the furnace residence time, the paint coating doesn't exhibit a melting point and that Fe from the steel substrate and oxygen from the furnace atmosphere diffuse into the paint coating, leading to changes in coating stoichiometry and morphology. Meanwhile, the pre-ceramic polymer undergoes pyrolysis, and the paint coating structure transforms from an amorphous phase to a crystalline phase.

The new coating showed considerable benefits in reducing manufacturing complexity and cost. It enables an optimized hot stamping process with lower furnace temperatures and residence time, eliminates the issue of coating buildup on the furnace rollers during heating and on the die cavities during forming, and results in lower maintenance costs for the hot stamping process.

The new coating opens opportunities for exploring new steel substrate alloy developments with lower austenitization temperatures and higher material strength and eliminates the concern of hydrogen embrittlement. Further reduction of manufacturing complexity and cost could be achieved by exploring additional heating technologies for hot stamping, such as resistive and induction heating.

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