Investigation of the impact of heat treatment on the layer formation of AlSi-coated boron-manganese steel

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Abstract. Hot stamping is a well-established technology for producing safety relevant car components. The usage of hot stamping allows for reduced sheet thicknesses and weight of formed parts due to an increase in strength because of martensitic phase transformations. To prevent surface carburization and the formation of oxide layers AlSi coatings are applied to the workpiece surface. Due to a lack of suitable lubricants at process temperatures of over 800 °C, severe friction and wear affect the final part quality. Furthermore, process temperatures influence the layer formation and surface roughness due to diffusion processes. Consequently, a deeper process understanding is needed to acquire detailed knowledge of the impact on the tribological behavior to increase part quality and reduce tool wear. To this end the influence of heat treatment parameters on the coating layer and the subsequent layer formation is analyzed by comparing the resulting material composition and surface roughness. This is achieved by analyzing the impact of the furnace temperature and holding time on an AS150 coating and determining the effect of the parameters via metallographic investigation and tactile measurement.

1. Introduction
Hot stamping of boron manganese steels is a technology used primarily in the production of safety relevant components for the modern car body-in-white like A- and B-Pillars, cross members and side sills [1]. The direct press hardening process combines forming at temperatures of 650 °C to 850 °C with hardening in a single process step [2], whereas the indirect route consists forming prior to the heat treatment, with only a calibration step included with the hardening [3]. Due to furnace temperatures of 900 °C to 950 °C and varying holding times dependent on sheet thickness, the workpiece material undergoes austenitization [4]. Simultaneous with the forming motion the workpiece is quenched and undergoes a phase transformation to martensite [5] with a cooling rate above 27 K/s, which results in tensile strengths of approximately 1500 MPa [6]. This allows for reduced sheet thicknesses with improved mechanical properties at the same time and for the production of lightweight safety relevant components. While 22MnB5 steels are commonly used in hot stamping, uncoated workpiece material will lead to decarburization and scale formation on the surface [7]. The oxide layer leads to a reduction in heat transfer rate between contact partners and damages the tool surface due to hard particles [8]. Furthermore, subsequent process steps like spot welding or finishes like painting are negatively affected due to the brittleness and high roughness of the oxide layer, which necessitates additional removal and cleaning processes for the part in the form of sand- or shotblasting [8]. To prevent scale formation and the negative impact of the oxide layer, hot stamping workpieces are coated. For the direct process route...
aluminium-silicon (AlSi) based coatings are commonly used [9]. Sherepenko et al. [10] investigated the impact of the parameters furnace temperature and holding time in the furnace on the coating layer for AlSi-coated 22MnB5 steels. They found both parameters to have a significant impact on the formation of the layer composition. While investigating the influence of the layer coating of 22MnB5 steels on subsequent spot weldability after heat treatment, Jüttner [11] came to the same conclusions for the furnace temperature and holding time. The coating layer undergoes a change in thickness as well as composition after passing through the furnace. Additionally Jüttner found the thickness of the coating layer in the initial state before heat treatment to have a significant influence [11]. Due to high process temperatures, lubricants cannot be used, which results in high friction and wear occurring in the process. While the layer coating formation has been investigated regarding weldability because of the coating layer, the impact on tribological properties due to layer coating and heat treatment have not yet been analyzed. Detailed knowledge between the cause and effect relation of material properties and the resulting tribological behavior is needed for the calculated design of properties of the final part. The goal within the scope of this paper is to analyze the influence of heat treatment parameters on the coating layer formation, identify the resulting phases and create a baseline for further studies on the impact of heat treatment parameters on tribological mechanisms and wear in the industrial hot stamping process.

2. Methodology and experimental setup

2.1. Parameters and methodology

The parameters for the heat treatment of the material were selected based on previous studies on the coating layer. Heating rate, furnace temperature and holding time in the furnace were selected to be analyzed in the study. Each parameter was varied at three industrial-related level settings (low, mid, high) and repeated three times, with the order of the experiments randomized to guarantee repeatability. The parameters and selected levels are listed below in table 1.

| Parameter                        | Levels            |
|----------------------------------|-------------------|
| Furnace temperature in °C        | 880; 910; 930     |
| Furnace duration in s            | 60; 270; 600      |
| Heating rate in K/s              | 5; 25; 50         |

After heat treatment the roughness Rz of the samples was determined via tactile measurement. The samples were then cold mounted and polished for micrograph analysis with a Keyence VK-X200 microscope. Using the micrograph image, the height of the coating layer could be determined as well as a qualitative analysis performed. To further identify the occurring phases in the coating layer, an image analysis approach using the open source software ImageJ was applied. In the first step the resulting micrograph image was manually edited to remove the cold mounting material as well as the boron manganese base material. The resulting modified image is then fed into the ImageJ software, where it can be classified via the Trainable Weka Segmentation plugin. With the plugin the user can create classes and manually select traces for each class. With the selection the plugin determines which pixels in the image can be assigned to which class based on similarity to the selected traces in each class. Using this method, the rest of the image is then categorized into the different classes. After the successful segmentation the result can be displayed as probability maps, in which the probability of each pixel belonging to a class can be presented as a separate black and white images. Using the probability maps, the number of pixels for each class can be counted and the total percentage of that class can be calculated in relation to the overall image size. The principle is demonstrated below in Figure 1 where the image was categorized and then split into three classes (Phase 1, Phase 2 and Background).
2.2. Material and experimental setup

For the workpiece a sample size of 70 mm x 50 mm was used with a boron manganese steel material of 22MnB5 and an aluminium-silicon based coating layer applied with a strength of 150 g/m². The coating has a wide application for sheet steels used in automotive manufacturing or heat shields and boilers due to its excellent resistance to both corrosion and elevated temperature oxidation [11]. The coating exhibits poor forming properties at room temperature and is mainly applied in the direct hot stamping approach [10]. Although process and furnace temperatures are above the melting temperature of AlSi at 620 °C, diffusion processes from the base material create an AlSiFe layer which consists of different intermetallic phases and prevents a melting of the layer [12]. The varying heating rate was realized on the thermomechanical simulator Gleeble 3500 GTC produced by Dynamic Systems Inc. The simulator runs a current through the sample thereby utilizing electrical resistance for heating. Due to this a temperature gradient develops in both width and length direction with the sample only being heated in the center. To monitor the temperature in the process, type K thermocouples are spot welded onto the sample. Further heat treatment experiments were conducted using an annealing and hardening furnace ME 87/13 by RHODE. The furnace can achieve temperatures of up to 1300 °C with KANTHAL heating elements mounted on support rods on three sides of the furnace walls, providing an even temperature distribution. Double layer insulation prevents heat escaping from the furnace surface while guaranteeing a homogenous furnace atmosphere for the heat treatment of the samples.

3. Results and discussion

3.1. Heating rate

The heating rate was investigated using the Gleeble 3500 GTC. The rates of thermal increase were realized at 5, 25 and 50 K/s reaching a temperature of 930 °C. The sample was then held at the set temperature for 60 seconds after which it was allowed to cool down to room temperature without a quenching process. Due to the small size of the heating spot in the center of the sample, no reliable roughness measurements could be taken. The micrograph image, as seen in Figure 2, shows the formation of a lighter grey coating layer interspersed with darker grey specks. Additionally, darker spots appear in the coating layer which can be due to the Kirkendall effect [12] and the diffusion of elements from the coating layer to the base material creating pores. At the edge area of the coating layer a darker border phase can be identified followed by the 22MnB5 base material. With an increase in heating rate to 25 K/s a visible rise in crack and pore occurrences can be observed. At even higher heating rates of 50 K/s the layer dissolution becomes more prevalent.
Figure 2. Coating layer phase formation at different heating rates

While an increase in the heating rate leads to a stronger layer dissolution, no visible impact on the formation of the darker spots can be observed. The main mechanism to prevent layer melting is based on diffusion processes between base material and coating, with the melting point of the AlSi coating at 620 °C. It can be concluded that higher heating rates lead to a melting of the coating layer due to the temperature being above the melting point of the current composition of the coating layer. This likely delays the diffusion processes and leads to the visible layer dissolution. The cracks can also be attributed to the melt pool due to the layer solidifying again after the process. This leads to the conclusion that the heating rate has no significant influence on the layer formation itself. Additionally, the heating rate of the furnace was determined to be at 5 K/s by spot welding type K thermocouples on a sample. Further heat treatment experiments were conducted using the annealing and hardening furnace to guarantee transferability of the findings to the industrial hot stamping process.

3.2. Furnace temperature and holding time
For the furnace temperature investigation the samples were placed in the ME 87/13 furnace at 880, 910 and 930 °C. Additionally, experiments were conducted at different holding times of 60, 270 and 600 seconds which was counted with the sample reaching the required furnace temperature. Each experiment setting was repeated 3 times with the samples being placed on brackets in the furnace to minimize direct contact and allow for a homogeneous heating process through the furnace atmosphere. The samples were manually removed after the heat treatment and allowed to reach room temperature with a cooling rate below 27 K/s and no quenching process and martensite transformation. The roughness measurements for the samples, as displayed in Figure 3, show no significant change in surface roughness due to furnace temperature.

Figure 3. Influence of heat treatment parameters heating duration and furnace temperature on surface roughness Rz
Similar to the furnace temperature, the heating duration exhibits no significant change in roughness at a heating duration of 60 seconds compared to 270 seconds. A further increase of the holding time to 600 seconds results in a significant increase of the surface roughness. The jump in surface roughness dependent on the time in the furnace can be ascribed to ongoing diffusion processes in the layer which are not finished at 60 and 270 seconds. The influence of the diffusion processes on the surface roughness Rz peaks at 600 seconds with 17.5 µm. Similarly to the roughness Rz, the height measurement performed on the micrograph images, as seen in Figure 4, show no significant difference in the layer height for the varying temperatures while a decreasing trend can be observed for increasing holding times.

![Image](https://example.com/image1)

**Figure 4.** Influence of heat treatment parameters heating duration and furnace temperature on layer height

The micrograph images of the samples clearly show the formation of different phases in the coating layer similar to the heating rate samples. For the identification of the phases, EDX measurements were performed for the various parameter settings. As seen in Figure 5, multiple phases could be identified which can be categorized into four general phases. Phase 1 initially consists of separate segments which gradually form into a thin line in the coating layer due to the influence of the heat treatment, as already seen in the heating rate micrographs (see Figure 2). It consists of roughly 32 % aluminum, 51 % iron and 12 % silicon. Phase 1 is embedded in the rest of the layer coating which was classified as Phase 2 and makes up the majority of the layer material. The composition of Phase 2 sees an increase in the aluminum content to 50 % compared to Phase 1. Additionally, both iron and silicon content slightly decrease to 45 % and 2 % respectively.

![Image](https://example.com/image2)

**Figure 5.** EDX measurement
Both phases show no significant change in composition regardless of parameter setting or position in the layer. The coating layer is followed by a border phase which separates the coating material from the base material. At a holding time of 60 seconds the border phase includes trace amounts of aluminum and silicon similar to Phase 1 while the base material consists of mainly iron with no AlSi content. With an increase in holding time the amount of aluminum in the border phase increases. At the same time the same measure points for the base material show an increase in aluminum and silicon. This leads to the conclusion that aluminum and silicon continue to diffuse into the base material with continued heating. The respective percentage of the phases 1 and 2 could be identified via the image analysis. For Phase 1 a significant increase can be identified at a furnace temperature of 880 °C, as seen in Figure 6. The same trend of an increase in Phase 1 can be observed at 930 °C.

![Figure 6. Influence of the holding time on the relative area of Phase 1](image1)

While the furnace temperature itself has no significant influence on the relative area of Phase 1, the image analysis shows an interdependency between the two parameters furnace temperature and holding time. An increase in furnace temperature has a diminishing effect on the increase of Phase 1 caused by a prolonged holding time and continuing diffusion of AlSi into the base material. Contrary to Phase 1, Phase 2 sees a decrease in the total area with an increase in holding time. The decrease at a furnace temperature of 880 °C, as seen in Figure 7, is significant from 60 to 270 seconds as well as at 600 seconds. Similar to the formation of Phase 1, the influence of the holding time on the area of Phase 2 is diminished by an increase in furnace temperature, with only a trend in the decrease due to the holding time visible.

![Figure 7. Influence of the holding time on the relative area of Phase 2](image2)
The diminishing area of Phase 2 correlates with the reduction in total layer height at higher holding times and can be only partially explained by the increase in the Phase 1 area. The micrograph images, as seen in Figure 8, show an increase in crack formation in the layer itself, with the porous surface near area partially fracturing and breaking off and thus further diminishing the Phase 2 area. This is likely due to the brittleness of AlFe particles, which form in Phase 2 [13], and high thermal loads due to prolonged exposure to high temperatures in the furnace during heat treatment.

The micrograph images also confirm the findings from the image analysis with no significant change visible in the thickness or quantity of Phase 1 at different furnace temperatures. The phase initially consists of single specks which form a discontinuous layer at a holding time of 60 seconds, while further increases in holding time lead to a growth and connection of the separated Phase 1 layer to form a semi-continuous line due to ongoing diffusion of Fe-particles into the coating layer. The findings are consistent with previous research into the topic [12].

4. Summary and outlook
The study presented in this paper investigated the formation of the layer coating of AlSi coated 22MnB5 steel under hot stamping conditions. The influence of different heat treatment parameters was investigated regarding layer formation in the AlSi coating. While a melting of the coating at increased heating rates can be observed, the parameter only delays the diffusion and has no effect on the phase formation itself. The furnace temperature was found to have no significant influence on the surface roughness Rz, coating height or the phase formation as well, while a correlating effect with the heating duration could be observed. The heating duration has a significant influence on the surface roughness Rz due to ongoing diffusion processes. The parameter also shows an influence on the layer height which diminishes with prolonged heating time. The cause has been identified by analysis of the micrograph images and image analysis to be the fracturing and partial breaking away of the coating layer at prolonged holding times. Contrary to previous investigations [11], the study presented here showed no increase in the layer height because of the heat treatment. This is due to the fact that the previous studies focus not only on the coating layer but also on the border phase as well, which sees an increase in height and size due to continuous diffusion of aluminum and silicon elements into the base material as was noted in this study. Since the actual height and size of border phase is only visible after quenching and martensite forming, it was not considered in this study since the focus lies in the effect of the coating layer during the forming process before martensite generation. A new methodology of analyzing the coating layer has been developed in the study, which allows for a quantitative analysis of the micrograph image regarding layer formation and parameter influence. Future research should focus on the influence of heat treatment parameters on friction and wear in experiments performed on strip drawing test rigs, which simulate the industrial process. The findings of this study lay the groundwork for further analysis on the impact of the layer formation and heat treatment parameters on friction and wear mechanisms in hot stamping and will help deepen the understanding of cause and effect relations in the process.

Figure 8. Micrograph images of samples held at 880 and 930 °C for 60 and 600 seconds
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