Inferring the surface roughness of Al-Si coated 22MnB5 steel using an in situ laser speckle characterization technique

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Abstract. Hot stamping of aluminium-silicon (Al-Si) coated 22MnB5 steel blanks is widely used in the automotive industry to produce light yet crashworthy parts. However, the coating melts at \textasciitilde577°C and transforms into a rough intermetallic layer as iron from the base steel diffuses towards the surface. The blank surface roughness impacts the radiative properties during heating as well as weldability, paint adhesiveness, and cooling rate during forming and quenching. This study pioneers the use of laser speckle patterns, caused by the constructive and destructive interference of collimated light reflected off the blanks, to infer the evolving surface roughness of Al-Si coated steel coupons in situ. The results reveal a significant increase in surface roughness once intermetallic compounds reach the surface and that higher furnace set-points produce rougher parts.

1. Introduction

To combat greenhouse gas emissions and improve fuel economy, many automotive manufacturers use thinner structural components made of stronger steels to lighten vehicles [1]. In particular, ultra-high strength steel (UHSS) parts made from aluminium-silicon (Al-Si) coated 22MnB5 steel are ubiquitous for reducing vehicle weight without sacrificing crash protection [2]. These UHSS parts are made through hot stamping, a process in which blanks are heated to \textasciitilde950°C in a long roller furnace to austenitize the steel and then transferred to a press where they are simultaneously formed and quenched [2]. The protective Al-Si layer is used to prevent the blanks from oxidizing and decarburizing in the furnace [3] while additionally providing moderate long-term corrosion resistance [4].

The Al-Si coating has a melting point of \textasciitilde577°C, which will cause a brief liquid phase during heating [5]. The coating then re-solidifies as it reacts with iron that diffuses from the substrate steel, forming a variety of intermediate intermetallic phases during the heating cycle [6]. After the steel is austenitized, the surfaces of the coated samples transform into a complex microstructure of various Al-Fe-Si and Al-Fe phases [7]. While the liquid phase of the coating can have detrimental effects on the furnace [8], its transformation also drastically alters the surface roughness of the steel.

Surface roughness is often quantified by the arithmetic average roughness,
\[
R_s = \frac{1}{L} \int_0^L |z(x)| \, dx
\]  

where \(z(x)\) is the difference between the profile height at location \(x\) and its overall mean height and \(L\) is the sampling length. Iron diffusion into the Al-Si coating significantly increases the surface roughness of the samples [9, 10]. Borsetto et al. showed that a longer holding time resulted in increased surface roughness and a higher holding temperature yielded samples with larger coefficients of friction [11]. Liang et al. found surface roughness to increase with higher dwell temperatures and heating rates, until reaching a maximum value at approximately 700°C [12]. Jenner et al. showed a distinct increase in the surface roughness of the samples shortly after the melting point of the coating as well as enhanced void formation within the coating at higher hold temperatures [13]. Conversely, they showed that a lower heating rate produced a rougher surface. Ghiotti et al. also found that lower soaking temperatures resulted in surfaces with higher friction coefficients once they had been quenched [14]. Barreau et al. investigated the surface roughness of hot stamping blanks with different heating rates and noticed a general increasing proportionality but could not identify a clear trend [15]. Finally, Podor et al. were able to estimate the topography of Al-Si samples using a high temperature environment scanning electron microscope and 3D image reconstruction based upon the 2D images to acquire height maps [16]. They discovered that the surface roughness of the samples remained relatively constant until the 675°C mark, where it increased greatly until 715°C. From 715°C to the holding temperature of 900°C, the roughness of the specimens decreased slightly.

The surface roughness of the blanks significantly affect various industrial processes that occur during and after heating, including paint adhesion [17], the interfacial heat transfer coefficient between the quenching die and the hot stamping blank [18], and weldability [19]. Furthermore, the evolving surface roughness of steel blanks greatly affects their radiative properties [9, 10], which impacts the temperature profiles of the blanks in the furnace.

Traditionally, surface roughness is evaluated using contact profilometry via a stylus instrument or with an optical profilometer, but these techniques are limited to \textit{ex situ} characterization. While useful, \textit{ex situ} measurements may be inaccurate due to phase transformations undergone by the surface as the samples are extracted from the furnace. Various optical procedures have been developed to produce non-contact, in-process methods of inferring roughness. One such procedure is the analysis of the speckle pattern produced when collimated laser light is reflected off a rough surface. The pattern consists of a series of bright and dark spots, as seen in figure 1, which is caused by the constructive and destructive interference of the scattered electromagnetic waves [20].

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{speckle_pattern}
\caption{Example of a speckle pattern.}
\end{figure}
While several statistical processes exist to quantify the relationship between speckle patterns and surface topography, many researchers have related the contrast of the speckle pattern, i.e. the standard deviation of the pixel intensities within the image, directly to the surface roughness [21, 22, 23]. Rougher surfaces will scatter collimated light to a greater degree, yielding speckle patterns with more frequent deviations from the mean intensity value and higher contrast parameters. Surface roughness is proportional to the contrast of the speckle pattern provided that

$$R_u \leq \frac{\lambda}{4\cos \theta}$$

(2)

where $\lambda$ is the wavelength of the laser and $\theta$ is the angle of incidence [21]. The correlation is invalid for rougher surfaces due to the enhanced scattering and resultant diffuse component of the laser illumination [24]. To evaluate rougher surfaces, Persson implemented a correlation method that compares two speckle patterns from the same surface at different angles of incidence and laser wavelengths [20].

Other researchers have employed the “bright-dark ratio method”, which has a larger measurement range. This procedure converts the speckle pattern into a binary image of bright, $B$, and dark, $D$, pixels based on one or more cut-off values. If the intensity of an individual pixel is greater than the specified threshold, it is counted as bright and vice versa. Kayahan et al. compared the ratio of $B$ and $D$ to surface roughness and found an exponentially decreasing relationship to roughness values of $3\lambda$ [24]. Meireles et al. found an exponential proportionality between $B/D$ and surface roughness values up to 12.75 $\mu$m for various surface types, but showed that the $B/D$ ratio increased with increasing surface roughness [25]. They attributed this to their measurement setup as the image plane was aligned normal to the specimen and not in the specular direction of the reflected laser light. Using a similar setup, Hamed et al. related the surface roughness of aluminum samples to the proportion of bright values within the speckle images and also discovered a proportional relationship between the $B/D$ ratio and surface roughness [26]. Xu et al. used a modification of this technique to yield linear relationships between the surface roughness of metals and the $B/D$ ratio [27]. Finally, Wong and Li used this technique to analyze the changing surface roughness of parts moving through a machining process [28].

This paper documents the first application of laser speckle analysis to investigate the evolving surface roughness of the Al-Si coated 22MnB5 steel within a furnace. A laser was shone onto the surface of coated blanks heated within a laboratory muffle furnace via its exhaust port and the reflected light was imaged onto a camera sensor. A measurement model is derived by comparing B/D values of images against ex situ contact profilometry measurements. The surfaces of the extracted samples were further characterized using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) to compliment the measurements. The speckle measurement model is then used to provide an in situ estimate of the surface roughness of the blanks during heating. These results provide important new insights into the evolving surface state of the steel and may improve the reliability of furnace modeling techniques and proceeding industrial processes.

2. Material and Methods

The experimental apparatus is shown in figure 2, which is built around a laboratory scale muffle furnace. An optical breadboard is custom fit around the exhaust port on the furnace roof, which provides optical access to the coupon. Light from a green laser diode (Thorlabs® CPS520) with a wavelength of 520 nm is imaged through the exhaust port onto the sample using a flat mirror. A portion of the reflected light from the sample is then projected onto the imaging chip of a Nikon® D7200 camera and the resultant speckle image was analysed using MATLAB® software. An image of the evolving surface state was taken approximately every two seconds.
Coupons consisted of 38 mm x 38 mm x 2.0 mm Usibor® 1500-AS150 blanks. The coupons had a nominal coating thickness of approximately 25 μm and a nominal mass composition of 90% aluminium and 10% silicon [29]. An example cross-sectional scanning electron microscope (SEM) image of the as-received coating is shown in figure 3. Small Si-aggregates are present in the Al-Si coating and a thin layer of Al₇Fe₂Si resulting from the Al-Si hot dipping process separates the base steel and coating. Samples were heated at set-points of 600°C, 700°C, 800°C, and 900°C. One sample for each furnace set-point was fitted with a k-type thermocouple to collect temperature data. For each test, the furnace was soaked for at least 5 minutes to ensure steady-state operation.

Figure 2. Speckle characterization experimental setup schematic.

Figure 3. SEM image of cross-section of as-received Al-Si coated 22MnB5 steel at 750x magnification.
Ex situ surface roughness was characterized principally using a contact profilometer (Mitutoyo-SJ-400), which was calibrated against an optical profilometer (WYKO NT1100), which is considered to be more accurate. Both instruments are separately calibrated using their own calibration samples. The contact profilometer results are consistently lower than those of the optical profilometer by approximately 0.4 μm, as seen in figure 4, but show identical trends. The deviation could be attributed to differences in instrument resolution or the fact that the optical profilometer derives the roughness values from 2D maps whereas the contact profilometer relies on 1D line profiles. Since the discrepancy is reasonably constant, the proceeding analysis will be conducted with only the contact profilometer. Roughness profiles oriented at 0°, 45° and 90° with respect to the sample are recorded and averaged to produce the overall $R_a$ value to account for any anisotropy at the surface of the specimens.

![Figure 4. Comparison of contact and optical profilometer roughness values.](image)

Finally, samples heated at the 600°C and 800°C set-points were removed from the furnace at intermediate heating times, immediately quenched with a stream of nitrogen gas, and then analysed using a SEM Zeiss Ultra Plus machine to assess intermetallic diffusion levels and chemical composition.

3. Results and Discussion

3.1 Speckle Pattern Characterization

To assess the validity of using speckle patterns to quantify the roughness of the blanks in situ, ex situ tests were conducted with various steel samples extracted at different furnace temperatures and dwell times to build a wide roughness sampling range. The $B/D$ ratio is sensitive to the cut-off parameter used to differentiate between the bright and dark pixels. We found that a threshold of 1.075 times the mean pixel intensity of the images yielded optimal results with respect to noise propagation for the in situ trials while giving a generally monotonic relationship between the $B/D$ ratio and surface roughness. An exponentially decreasing trend between the $B/D$ ratio and the roughness of the surface can be seen in figure 5. The best fit quadratic curve shown in figure 5 was then used to generate the in situ roughness profile in the following section.
3.2 In Situ Analysis

The heating profiles for the different furnace set-points are displayed in figure 6a. Various inflection points in these curves can be detected, which is visualized in figure 6b when the heating rate is plotted alongside the temperature profile. The temperature derivative, calculated through first-order central finite differences, shows the melting point of the coating as well as the onset (T_{Ac1}) and conclusion (T_{Ac3}) of the austenitization process, aligning with the results of Jhajj et al. [30].

An in situ B/D profile for a sample heated to 800°C is shown in figure 7. After approximately 20 seconds in the furnace, a distinct peak can be seen in the B/D plot, indicating a decrease in surface roughness and implying the coating has melted. Furthermore, a sharp decrease and subsequent increase can also be seen in the B/D signal shortly thereafter, before decreasing and finally plateauing. This closely resembles previously-reported in situ reflectivity measurements, which was hypothesized to be caused by an intermittent oxidation reaction between the two peaks [31]. Using in situ SEM images of the evolving surface state of hot stamping coupons, Barreau et al. hypothesized that an intermittent melting reaction occurs at approximately 655°C, potentially explaining the existence of the second peak [15].
Notably, none of the samples heated to 600°C showed the two-peak behaviour, which echoes reflectance measurements from our previous study [31]. While the absence of the second peak was attributed to the existence of a “mushy” region on the Al-Si binary phase diagram, it is more likely that the samples simply do not reach the temperature necessary to incur the secondary melting reaction specified by Barreau et al. [15]. While the two-peak behaviour was detected for many of the individual trials heated at furnace set-points above 600°C, it was not present in every case. This could be because of the relatively low temporal resolution of the camera. To represent multiple trials, the mean of 5 $B/D$ plots was taken to build an average curve for each temperature along with confidence intervals. An example of this process using the 700°C trials is shown in figure 8.

Using the relation given in figure 6 and the average of 5 $B/D$ curves, in situ roughness profiles were generated for samples heated at the different temperature set-points, as seen in figure 9. The different trials all exhibit an abrupt increase in roughness once the Al-Fe-Si compounds diffuse to the surface of the coating, and the final roughness increases with increasing furnace set-point. Samples were then extracted from the furnace and analyzed with the contact profilometer. The square data points in figure
show the average of four samples after a complete heating cycle at each of the four temperatures. In the case of samples heated to 900°C, the average roughness was 3.12 µm; accounting for the average 0.4 µm difference between the contact and optical profilometer, this result is consistent with roughness values of approximately 3.40 and 3.50 µm, reported by Shi et al. [9, 10] and Podor et al. [16] for coupons heated under similar conditions.

Furthermore, circular data points signify the average of two samples that were intermittently removed from the furnace at various dwell times and immediately quenched with a stream of nitrogen gas to preserve the surface microstructure. These samples were then measured with the profilometer and mostly show higher roughness values than what the B/D model predicts. However, this result is reasonable, given that any liquid constituent present at the surface of the samples would solidify when quenched, altering the overall roughness.

![Figure 9. In situ roughness profiles for furnace set-points of 600°C, 700°C, 800°C and 900°C with ex situ contact profilometry measurements.](image)

3.3 Ex situ SEM and EDS Analysis

The SEM and EDS analysis provided herein is from our previously published work [31]. The overall surface chemical composition measured using EDS over a 150 µm × 100 µm area for samples heated at set-points of 600°C and 800°C for various furnace dwell times can be seen in table 1. At both set-points, the increasing oxygen content implies the formation of an oxide layer. Additionally, rising iron levels indicate iron diffusion from the base steel to the outer layer. These results are consistent with the findings of Shi et al., as they detected alumina and iron oxides at longer heating durations [9, 10].

Backscattered SEM images of the sample surfaces can be seen in figure 10. The presented phases were approximated through the elemental compositions given by the EDS analysis. The propagation of Al₄.5SiFe can be detected at both furnace set-points, aligning with the results of Grigorieva et al. [7]. Si-rich aggregates reported by Borsetto et al. [11] are present in the shorter dwell time samples but disappear when subjected to longer heating durations. Moreover, the surface also appears to become smoother in figure 10f compared to figure 10e, corresponding to the drop in the B/D signal between the two peaks, potentially caused by the intermittent melting reaction proposed by Barreau et al. [15].
Table 1. Surface chemical composition by furnace set-point and dwell time.

| Furnace Set-point (°C) | Dwell Time (s) | Chemical Composition (at. %) |
|------------------------|----------------|-----------------------------|
|                        |                | Al  | Fe  | Si  | O   |
| 600                    | 150            | 81.8| 0.39| 14.4| 3.41|
|                        | 210            | 86.8| 1.03| 8.37| 3.76|
|                        | 420            | 86.5| 2.31| 2.46| 8.70|
|                        | 40             | 86.4| 0.57| 11.8| 1.21|
|                        | 50             | 84.9| 1.01| 11.8| 2.28|
| 800                    | 80             | 77.6| 0.7 | 11.4| 10.3|
|                        | 100            | 80.3| 0.75| 11.4| 7.61|
|                        | 180            | 78.1| 1.38| 10.2| 10.3|

The evolution of the coating was further analysed using backscattered SEM cross-section images along with EDS line scans through the coating, as seen in figure 11. These images show progressive iron diffusion from the base metal to the outermost layer and a variety of Al-Fe-Si and Al-Fe intermetallics, including Al$_4$FeSi, Al$_7$Fe$_2$Si, and Al$_5$Fe$_2$. Iron diffusion occurs at a greater degree at the higher furnace set-point and the samples held at the longest dwell times show intermetallic layers reaching the surface, aligning with the final increase in surface roughness for the different trials seen in figure 9. Grigorieva et al. also identify Al$_7$Fe$_2$Si and Al$_5$Fe$_2$ layers after the melting of the coating [7]. Additionally, Borsetto et al. show similar intermetallic compounds in samples heated to 600°C [11]. Heightened oxygen content at the surfaces of the samples heated to the longer dwell times further imply the formation of an oxide layer.
Figure 10. Backscattered SEM images depicting Al-Si coating evolution at the surface after heating at 600°C for: (a) 150 s, (b) 210 s and (c) 420 s; and at 800°C for (d) 40 s, (e) 50 s, (f) 80 s, (g) 100 s and (h) 180 s. (500x magnification).
Figure 11. Back-scattered SEM cross-section images and EDS line scans showing coating evolution after heating at: (a) 600°C for 420 s; and 800°C for (b) 50 s and (c) 180 s (750x magnification).

4. Conclusions
The evolving surface roughness of Al-Si coated 22MnB5 steel significantly effects several hot stamping operations and must be understood to improve part quality and industrial processes. This work pioneers the application of speckle analysis for in situ roughness measurements on the intermetallic coating. These measurements are valuable for process control and optimization, and also provide important insight into the evolution of the coating within the furnace. Measurements conducted on samples heated above 600°C indicate abrupt changes in roughness preceding the melting of the coating, which may be linked to the formation of an intermittent oxide layer and a secondary melting reaction. Longer hold times show a dramatic increase in the surface roughness followed by a stable value once the evolution
of the coating was complete. Higher furnace set-point temperatures produced rougher surfaces due to the increased diffusion occurring within the coating. Ex situ scanning electron microscopy and energy dispersive X-ray spectroscopy confirmed that iron diffusion towards the surface occurred quicker and to a greater degree with longer heating durations and higher furnace set-points. These methods also indicate a distinct change in surface composition shortly after the melting of the coating and increased oxygen content, pointing to the formation of an oxide layer.

Future work will focus on adapting this technique and creating a diagnostic tool to assess surface roughness within industrial furnaces. Furthermore, the growth of the oxide layer and the reactions occurring within and at the surface of the coating will be characterized using in situ Fourier transform spectroscopy measurements and Raman microscopy, both of which can detect the presence of oxides and intermetallic phases based on their various bond energies. Finally, the effect of coating weight on the in situ radiative properties of the steel will be investigated.

5. References
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