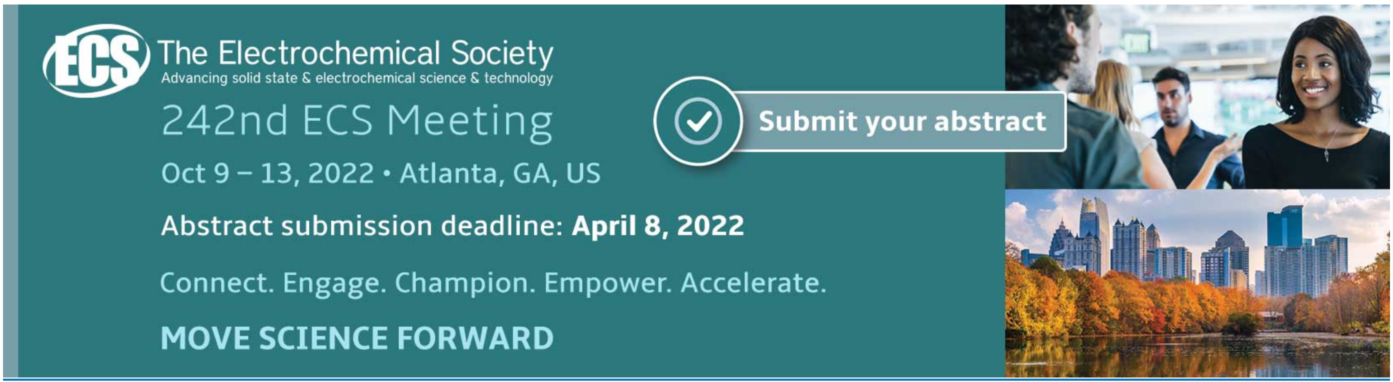
IOP Conference Series: Materials Science and Engineering

Effect of heating temperatures on AlSi coating microstructure and fracture during hot-tensile tests

To cite this article: S B Zaman *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1157** 012018



Effect of heating temperatures on AlSi coating microstructure and fracture during hot-tensile tests

S B Zaman[1], **J Hazrati**[1], **M B de Rooij**[2] **and A H van den Boogaard**[1]
[1]Nonlinear Solid Mechanics, Faculty of Engineering Technology, University of Twente, Enschede, The Netherlands
[2]Surface Technology & Tribology, Faculty of Engineering Technology, University of Twente, Enschede, The Netherlands

E-mail: `s.b.zaman@utwente.nl`

Abstract. In this article, the fracture behavior of different AlSi coating micro-structures is investigated. By changing the heating temperature, different AlSi coating micro-structures are obtained, due to varying diffusivity of iron. To study the fracture behavior of different coating micro-structures, uniaxial tensile tests are conducted at 700 °C. The hot tensile test involves heating the as-coated press hardening steel in a furnace to heating temperatures of 750, 920 and 1000 °C for 6 minutes of dwell time, after which the sample is cooled to the deformation stage at 700 °C. In this stage, the samples are uniaxially deformed for a fixed 20% macroscopic strain followed by cooling to room temperature. After the test, the coating micro-structure and fracture pattern are inspected under the microscope. For samples heated to 750 and 920 °C, coating fracture is observed; however, for samples heated to 1000 °C, no coating fracture is observed during the tensile deformation at 700 °C. The AlSi coating micro-structure, after heating at 1000 °C becomes sufficiently ductile to withstand 20% strain at 700 °C. The same micro-structure, however, fractures during uniaxial tensile deformation at 600 °C.

1. Introduction

Hot stamping is a process which combines the heat treatment and deformation at elevated temperatures. During this process, steels are susceptible to decarburization and oxidation, both of which hamper its quality and strength. Therefore steel sheets are generally coated prior to hot stamping. In particular, for press hardening steels (PHS), aluminum with 10 wt.% silicon (AlSi) coating is generally used for the hot stamping operation. By generating a dense alumina scale on the surface, an oxidation-resistant coating layer is obtained [1, 2]. Apart from its ability to prevent oxide scales on the surface, the AlSi coating reduces friction between the contacting surfaces [3]. However, during deformation at high temperatures, it has been reported that the AlSi coating layer fractures, resulting in tool wear and an oxidized substrate [4, 5].

1.1. Evolution of AlSi coating micro-structure

Hot stamping involves heating above the austenitization temperature followed by simultaneous deformation and quenching. In the case of an AlSi coating, the heating stage defines its overall mechanical behavior [6]. During heating above 600 °C, Al–Si alloy in the coating melts and iron from the substrate diffuses into the coating, leading to the formation of different Fe–Al–Si intermetallic compounds [7]. Due to Fe-diffusion, although various other compounds are formed in the AlSi coating, the intermetallics of Fe_2Al_5 and FeAl stabilize and predominate in greater fractions than any other Fe–Al–Si intermetallics [8–10]. Furthermore, by changing the heating rate, temperature and dwell time, different coating micro-structures are generated. In particular, the heating temperature is significant because it controls the rate of Fe-diffusivity in the AlSi coating from the substrate steel. A controlled heating stage may generate a coating micro-structure which deforms under uniaxial tensile loading condition without causing fracture.

1.2. Objective of this study

The goal of this study is to experimentally investigate the fracture behavior of different AlSi coating micro-structures under uniaxial tension at 700 °C. For this purpose, hot tensile experiments are conducted on 3 distinct coating micro-structures, resulting from different heating temperatures. After straining, the fracture behavior of different AlSi coating micro-structures is compared using microscopy techniques, including SEM–EDS measurements, to verify the chemical composition of each coating micro-structure.

2. Experiments

AlSi-coated PHS is used for this study. Before the test, the coated sheet material is 1.5 mm thick, while the AlSi coating is 25–30 μm thick on both sides. Uniaxial tensile samples are made via laser cutting technique, maintaining a fixed gauge width to length ratio of 1:4. Temperature-resistant type K thermocouples are attached to the sample shoulders, ensuring that the gauge section is unaffected from the adverse effects of spot-welding on the AlSi coating. To conduct hot-tensile tests, a tensile fixture with ceramic blocks at the gripping edge is manufactured. Figure 1 shows the experimental setup under operation at elevated temperatures.

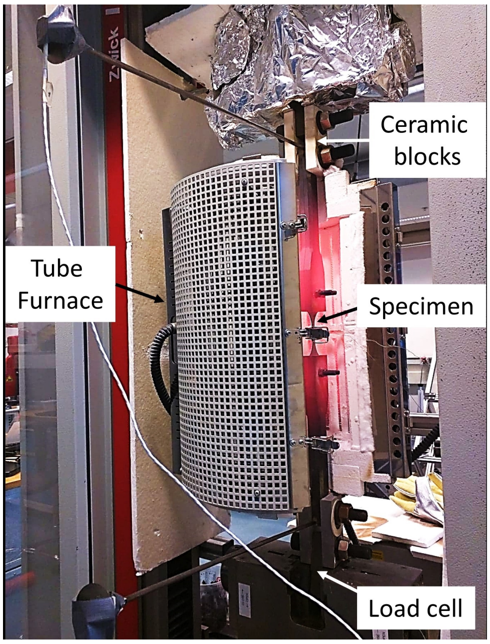


Figure 1. The hot-tensile experimental setup.

2.1. Evolution of AlSi coating micro-structure during heating

The heating stage determines the mechanical behavior, in particular, the intermetallic distribution in the coating layer. Based on the Fe-content, the ductility of AlSi coating is determined. Different coating micro-structures can be obtained, by changing the heating temperature while keeping the heating rate and dwell time fixed at 0.5 K/s and 6 minutes, respectively. Figure 2(a) shows the evolution of AlSi coating micro-structure at a heating temperature of 750 °C. In this case, due to Fe-diffusion, Fe–Al intermetallics are generated in the coating layer. The SEM–EDS inspection shows presence of Fe_2Al_5, with significantly large presence of Si at the center of AlSi coating layer. Since higher thermal energy is required for the growth of FeAl than of Fe_2Al_5, FeAl starts to form in the coating only when the heating temperature exceeds 900 °C, especially at the location of high Si content [11]. Regarding the coating–substrate interface, after heating at 750 °C for 6 minutes, the interface is discontinuous, with a sharp transition in compositions between Al (in the coating) and Fe (in the substrate). Regarding the void distribution after heating at 750 °C, the large voids are situated near the surface while the small ones are situated near the coating–substrate interface (Fig. 2(a)). Although such a low heating temperature is not industrially relevant due to the possibility of incomplete austenitic transformation in the substrate, it is useful in understanding the evolution of the AlSi coating micro-structure.

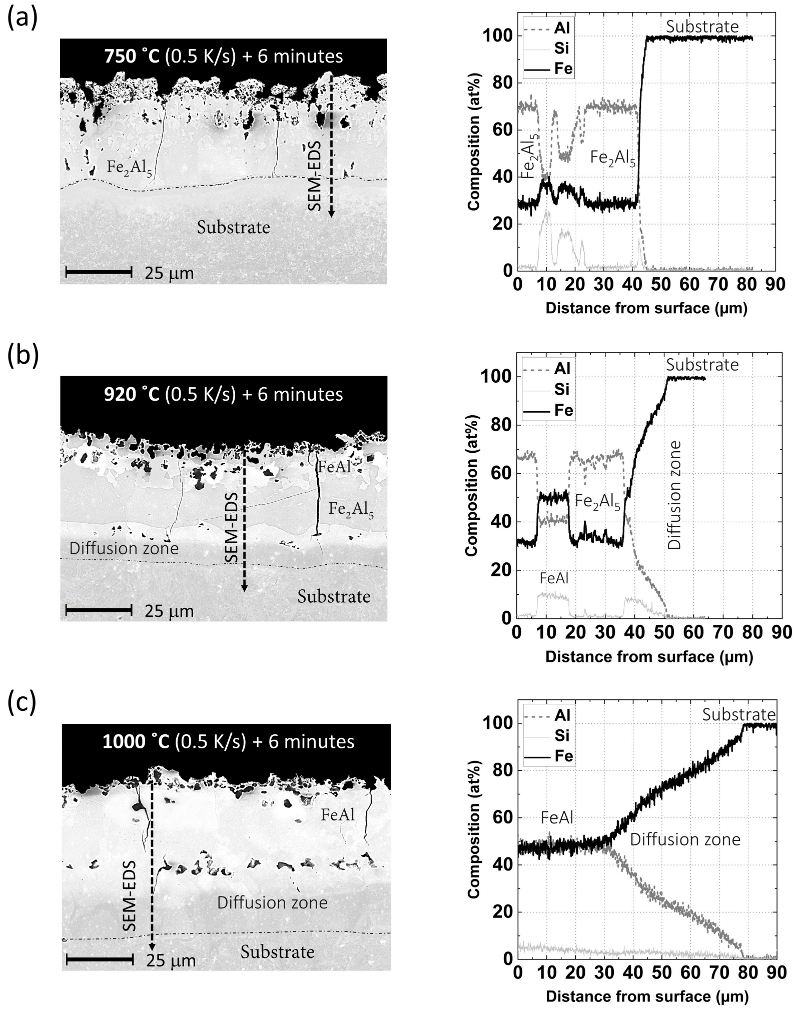


Figure 2. The AlSi coating micro-structure and atomic composition of AlSi coating after heating AlSi-coated steel at (a) 750, (b) 920 and (c) 1000 °C for 6 minutes of dwell time.

After heating the coated PHS with standard heating parameters (i.e., at 920 °C for 6 minutes of dwell time), the AlSi coating micro-structure evolves to various Fe–Al intermetallic compounds (Fig. 2(b)). According to the SEM–EDS measurements, the AlSi coating is dominated by layers of FeAl, Fe_2Al_5 and diffusion zone (α-Fe). Regarding the void distribution, although Kirkendall voids are small and restricted within the diffusion zone, large voids are found near the coating surface. Although high heating rate ($\sim$10 K/s) is maintained in practice, a low heating rate of 0.5 K/s in the setup did not significantly change the micro-structure as the AlSi coating is still dominated by the same Fe–Al intermetallic compounds.

After heating the AlSi-coated PHS at a temperature of 1000 °C for 6 minutes of dwell time, a drastic upsurge in the rate of Fe-diffusion is observed, increasing not only the thickness but also the amount of Fe-rich intermetallics in the coating layer (Fig. 2(c)). Due to increased rate of Fe-diffusion, the diffusion zone grows in size, transforming the 30 μm virgin coating to an 80 μm thick layer. According to SEM–EDS measurements, half of the coating is dominated by the diffusion zone while the other half is composed of FeAl. Regarding the void distribution, the Kirkendall voids exist between the layers of FeAl and diffusion zone. In contrast, the Kirkendall voids after heating at 1000 °C are significantly larger than what is observed at 750 or 920 °C. This means that the void size is dependent on the thermal history of the experiment. To sum up, the heating temperature significantly evolves the coating micro-structure, favoring the formation of Fe-rich compounds with rise in temperature level.

2.2. Investigating AlSi coating fracture during hot tensile tests

After heating the AlSi-coated PHS dog-bone samples at different heating temperatures followed by 6 minutes of dwell time, they are cooled to 700 °C where uniaxial tensile deformation is conducted to a fixed 20% macroscopic strain. From SEM–EDS atomic composition measurements, it is shown that the heating temperature plays an important role in terms of coating micro-structure, which is passed on to the deformation stage. To check the fracture behavior of these different coating micro-structures, all the deformation stage parameters; for instance, the strain rate and strain level, including the cooling rate during quenching, are kept constant. In this section, the ductility of 3 different AlSi coating micro-structures is tested at 700 °C. After the test, the deformed samples are inspected under the microscope to observe the distribution of coating cracks.

Figure 3 shows the distribution of coating cracks during tensile test at 700 °C, after being heated to 750 °C for 6 minutes. Since the heating temperature of 750 °C gives rise to a brittle Al-rich coating micro-structure (Fig. 2(a)), the cross-section view of the deformed sample shows mode-I coating fracture (Fig. 3(b)). It is possible that the lack of diffusion zone at the coating–substrate interface also increases the likelihood of mode-II interfacial fracture, shown in Figure 3(b). Such a coating fracture is considered severe because it detaches the coating layer from the substrate.

In Figure 4, after heating the coated PHS at 920 °C for 6 minutes, it is then cooled to 700 °C for uniaxial tension until 20% macroscopic strain. After the test, although the crack distribution from the top surface looks very similar to that in Figure 3(a), the cross-section view of the sample shows no mode-II interfacial fracture (Fig. 4(b)). Furthermore, due to the presence of ductile FeAl intermetallic compound, including a diffusion zone (Fig. 2(a)), the initiation of coating fracture is delayed compared to the micro-structure in Figure 3.

Interestingly, the samples heat-treated at 1000 °C for 6 minutes show no coating fracture during tensile test at 700 °C (Fig. 5(a)). At this heating temperature, since the AlSi coating fully transforms to FeAl (Fig. 2(c)), the overall coating, despite having large Kirkendall voids, is able to deform uniaxially for 20% macrscopic strain without causing coating fracture. Although micro-cracks are seen along the cross-section of AlSi coating (Fig. 5(b)), these are found in all the tested samples and are most likely generated during the quenching stage [14].

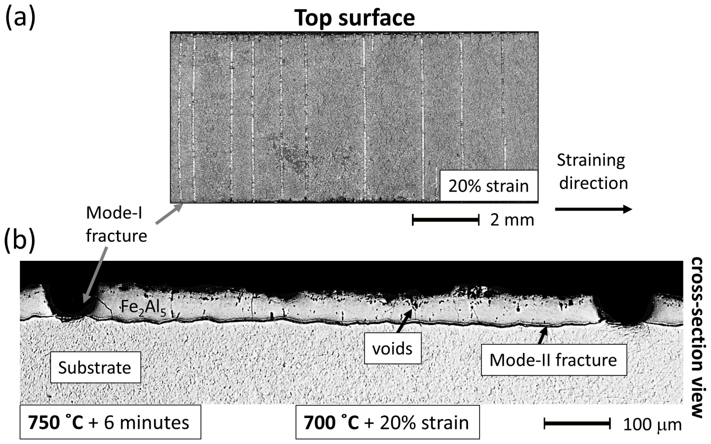


Figure 3. (a) Top and (b) cross-section optical inspections of the AlSi coating. The AlSi-coated steel is deformed at 700 °C after heating at 750 °C for 6 minutes of dwell time.

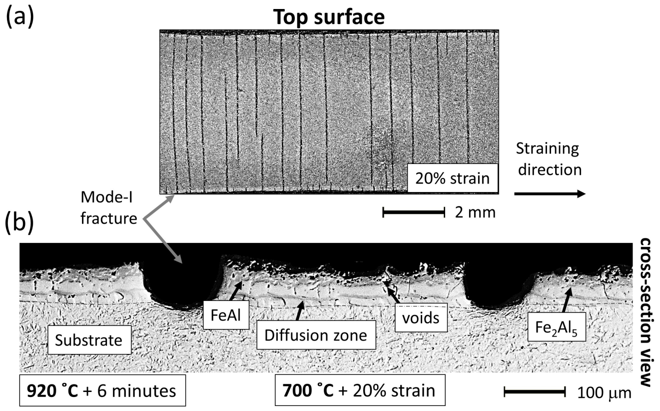


Figure 4. (a) Top and (b) cross-section optical inspections of the AlSi coating. The AlSi-coated steel is deformed at 700 °C after heating at 920 °C for 6 minutes of dwell time.

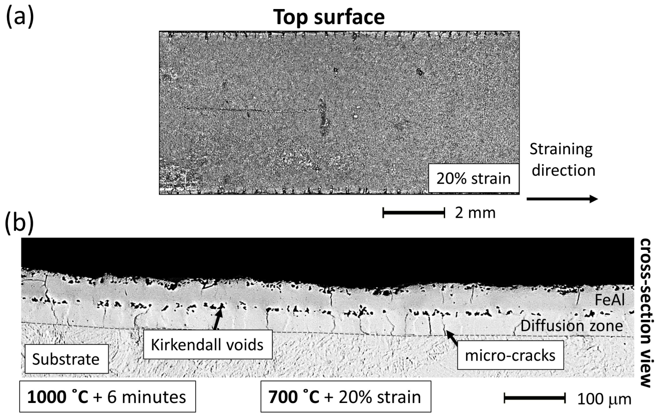


Figure 5. (a) Top and (b) cross-section optical inspections of the AlSi coating. The AlSi-coated steel is deformed at 700 °C after heating at 1000 °C for 6 minutes of dwell time.

In order to further investigate the fracture behavior of the Fe-rich coating micro-structure in Figure 2(c), the heating profile of 1000 °C for a dwell time of 6 minutes is repeated for a uniaxial tensile deformation at 600 °C. Figure 6 shows the coating crack distribution along the gauge section of tensile sample during the uniaxial tensile deformation at a temperature of 600 °C. At this deformation temperature, the coating cracks are visible from the top surface, showing significantly more cracking than at 700 °C. Upon cross-sectional inspection of the coated samples, the mode-I coating fracture is observed to penetrate through both the FeAl and diffusion zone, thus exposing the (steel) substrate (Fig. 6(b)). Although the AlSi coating after heating at 1000 °C does not show fracture during deformation at 700 °C, the strain localization around the Kirkendall voids is severe enough to cause coating fracture at 600 °C. This further confirms that the fracture behavior of AlSi coating is dependent on the thermal history prior to deformation. Furthermore, these mode-I coating cracks are most likely to have initiated at the onset of uniaxial tensile straining [15].

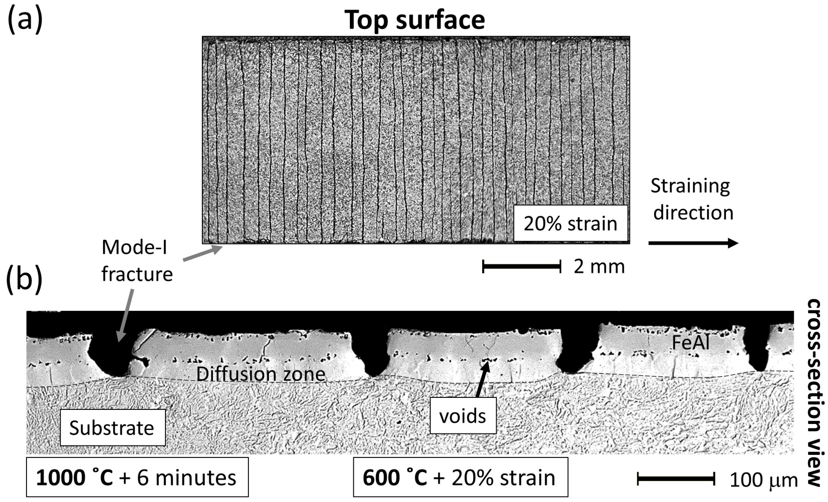


Figure 6. (a) Top and (b) cross-section optical inspections of the AlSi coating. The AlSi-coated steel is deformed at 600 °C after heating at 1000 °C for 6 minutes of dwell time.

3. Conclusion

This article investigates the fracture behavior of different AlSi coating micro-structures during uniaxial tensile tests at high temperatures. Based on the experimental results, the following conclusions can be made:

- After heating to 1000 °C (0.5 K/s) for a dwell time of 6 minutes, no coating fracture is observed when the AlSi-coated steel is uniaxially deformed at 700 °C for 20% macroscopic strain. The same micro-structure, however, fractures during identical tensile load case at 600 °C.
- At heating temperatures of 750 and 920 °C, mode-I coating fracture occurs during uniaxial tensile test at 700 °C.
- Fe-content of the complete coating determines the coating's fracture behavior during tensile deformation of AlSi-coated steel.

Acknowledgment

This research was carried out under project number S22.1.15583 in the framework of the Partnership Program of the Materials innovation institute M2i (www.m2i.nl) and the Technology Foundation TTW (www.stw.nl), which is part of the Netherlands Organization for Scientific Research (www.nwo.nl). The authors would like to thank TATA Steel for their materials and scientific feedback.

References

[1] Eggeler G, Auer W and Kaesche H 1986 *Journal of Materials Science* **21** 3348–3350
[2] Pint B, Zhang Y, Tortorelli P, Haynes J and Wright I 2001 *Materials at high temperatures* **18** 185–192
[3] Hardell J, Kassfeldt E and Prakash B 2008 *Wear* **264** 788–799
[4] Gui Z X, Wang K, Zhang Y S and Zhu B 2014 *Applied surface science* **316** 595–603
[5] Venema J, Hazrati J, Matthews D, Stegeman R and Van Den Boogaard A 2018 *wear* **406** 149–155
[6] Jenner F, Walter M E, Iyengar R M and Hughes R 2010 *Metallurgical and Materials Transactions A* **41** 1554–1563
[7] Liang W, Tao W, Zhu B and Zhang Y 2017 *Science China Technological Sciences* **60** 1088–1102
[8] Windmann M, Röttger A and Theisen W 2013 *Surface and Coatings Technology* **226** 130–139
[9] Sasaki T and Yakou T 2006 *Surface and Coatings Technology* **201** 2131–2139
[10] Cheng W J and Wang C J 2011 *Applied Surface Science* **257** 4663–4668
[11] Zhang N, Hu Q, Yang F, Lu W, Ding Z, Cao S, Yu L, Ge X and Li J 2020 *Metallurgical and Materials Transactions A* **51** 2711–2718
[12] Fu C, Ye Y Y, Yoo M and Ho K 1993 *Physical Review B* **48** 6712
[13] Fähnle M, Mayer J and Meyer B 1999 *Intermetallics* **7** 315–323
[14] Zaman S B, Hazrati J, de Rooij M, Matthews D T A, Venema J and van den Boogaard T 2018 *Forming Technology Forum* pp 87–92
[15] Zaman S B, Hazrati J, de Rooij M and van den Boogaard T 2020 *Procedia Manufacturing* **47** 602–607