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Investigation of the Effects of Hot Dip Aluminizing on Microstructural and Tribological Properties of Dual Phase (DP800) Steels

Sıcak Daldırma Alüminyumlama işleminin Çift Fazlı (DP800) Çeliklerin Mikroyapısal ve Tribolojik Özelliklerine Etkilerinin İncelenmesi

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Abstract

In this study, the effects of hot-dip aluminizing (HDA) (90 sec at 725°C) and post-heat treatment on the microstructural and tribological properties of DP800 steel were investigated. After HDA treatment, one of the sample was left to cool in air (HDA-1) and the other one left to cool in air after being subjected to a heat treatment for 1 hour in an oven set at 300°C (HDA-2). Evolution in mechanical properties depending on the developments in the coating microstructure investigated in terms of hardness and wear behaviour. Characterization results show that heat treatment increases the hardness value of hot dip aluminized steel to 1100 HV values. Considering the wear ball volume and friction coefficients, the most advanced tribological properties were observed in the heat-treated HDA process (HDA-2).

Key Words: DP800 steel, Hot-dip aluminizing, Heat treatment, Wear

Öz

Bu çalışmada, sıcak daldırmalı alüminyumlama (HDA) (725°C'de 90 saniye) ve ardıl ısıl işlemin DP800 çeliğinin mikroyapısal ve tribolojik özellikleri üzerindeki etkileri incelenmiştir. HDA işleminden sonra numunelerden biri havada (HDA-1), diğeri ise 300°C'ye ayarlanmış etüvde (HDA-2) 1 saat ısıl işleme tabi tutularak havada soğumaya bırakılmıştır). Kaplama mikroyapısındaki gelişmelere bağlı olarak gelişen mekanik özellikler sertlik ve aşınma davranışı bakımından incelenmiştir. Karakterizasyon sonuçları, ısıl işlemin sıcak daldırma alüminize çeliğin sertlik değerini 1100 HV değerlerine çıkardığını göstermektedir. Aşınan bilye hacmi ve sürtünme katsayıları dikkate alındığında en gelişmiş tribolojik özellikler ısıl işlem görmüş HDA prosesinde (HDA-2) gözlenmiştir.

Anahtar Kelimeler: DP800 çelik, Sıcak daldırma alüminyumlama, Isıl işlem, Aşınma

1 Introduction

Dual phase (DP) steels, which contain of ferrite and martensite phases, belong to the family of advanced high strength steels (AHSS) [1] [2] [3]. These steels, especially used in automotive applications, are preferred due to their good ductility and high strength properties. Namely, the dispersed hard martensite in the microstructure provides high strength, while the soft ferrite as a main phase in the microstructure provides good ductility and formability [3]. Since it is essential to protect the car body from corrosion and deterioration in the automotive industry, various commercial coating methods, such as hot-dip galvanizing (HDG, pure zinc), galvannealed coating (GA, zinc-10% iron alloy) and galfan coating (ZA, zinc-5% aluminum alloy), are applied to DP steels [4][5] . While zinc coatings are a preferred material for providing sacrificial cathodic protection to thin sheet automotive steels, they tend to create challenges during welding [6] [7]. Specially, the coating prone to vaporize aggressively due to the high heat input related to conventional welding methods (i.e., gas metal arc welding/GMAW) owing to zinc's low melting point (420°C) and boiling point (907°C). Consequently, standard welding techniques, such as GMAW, can result in near-joint problems such as porosity, blowholes, and zinc burn-off, making the welded area susceptible to corrosion. [8]. It has been found recently that Zn coatings cause liquid metal embrittlement (LME) in fusion welds produced using various techniques [9][10]. Such problems have prompted researchers to study with some other type of hot-dip coatings on DP steels. Hot dip aluminizing technique is a practical and cost-effective coating method, which is based on immersing the substrate material in molten aluminum alloy for a specified durations and forming an aluminide layer in the outer layer and a continuous interdiffusion zone at interlayer. Takata et al. [11] fabricated the Al–8.2Mg–4.8Si alloy-coated dual phase steels using hot-dipping. They reported that hot-dip Al-based coating is effective method for coating dual-phase steels with a controlled microstructure. Dede [12] studied hot-dip aluminizing (HDA) of dual phase steel. He reported that the coating formed with different HDA parameters did not cause a significant change in the mechanical and microstructural features of dual phase steel. Considering the studies on HDA are examined in the literature, it is seen that these studies are mainly on low carbon and alloy steels. Samsu et al. [13] studied the morphological and microstructural properties of the hot-dip aluminized carbon steel. They found that the intermetallic layer with high hardness obtained by the HDA process was thick and exhibited a fingerlike growth. Huilgol et al. [14] indicated that the formation of $FeAl_3$, Fe_2Al_5 and Al_7Cr phases during HDA of AISI 321 stainless steel in a pure Al bath. It was also reported the major intermetallic phases found on hot-dip aluminized steel were Fe_2Al_5 and $FeAl_3$ and these phases, which are brittle in nature, capable to transform into relatively ductile Fe-rich phases such as FeAl and Fe_3Al by a secondary diffusion treatment [15].

Inspired by these studies, this work investigated the effect of hot-dip aluminizing on the microstructural, hardness and

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tribological properties of DP800 steel. The samples (HDA-1) that were left to cool directly in air after HDA or that (HDA-2) were left to cool after a certain period of heat treatment in the furnace were characterized in detail. The purpose of applying heat treatment after HDA is to examine how a post-heat treatment causes a change in the coating layer and its effects on hardness and wear resistance, compared to the air cooling process after hot dipping, which is preferred in industrial applications. The application of aluminized coating on the steel surface not only provides corrosion protection, but also causes a decrease in surface hardness. In order to increase the application limit within the scope of the tribological properties of the HDA process, the post-diffusion process, which is thought to change the coating characteristic, has been applied.

2 Material and methods

2.1 Substrate preparation

Samples (DP800 steels) with a dimension of 20 mm × 20 mm × 2 mm were cut an abrasive cutter. Following cutting, the samples were ground using conventional metallographic processes using SiC papers 1200 grid size, cleaned ultrasonically with acetone, and then allowed to air-dry.

2.2 Coating procedures

The hot-dipping operation was conducted for 90 seconds in a bath of molten 99.9% pure aluminum in a graphite crucible set in a resistance furnace at 725°C. While some of the samples were left to cool in air after hot-dipping process, others were kept in a 300°C oven for 3600 s after dipping and then left to cool in air. The general process flow chart is in figure 1.

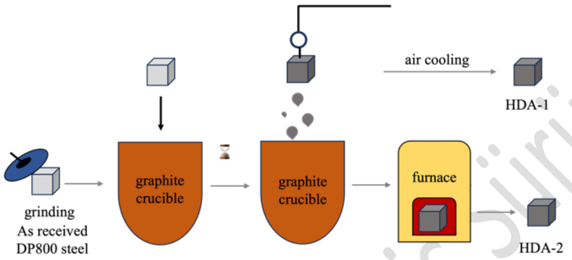

Figure 1. DP800 steel hot dip aluminizing process flow chart

2.3 Characterization

The microstructure was studied using a scanning electron microscope (SEM) (Carl Zeiss Gemini 300) integrated with energy dispersive X-ray spectroscopy (EDX) (Bruker XFlash 6I100). For phase analysis, samples were diffracted using an X-ray diffractometer (XRD, Bruker-AXS D8 Discover, 35kV and 28.5mA, CuKα radiation, 2θ range 20–90°).

The hardness tests of the samples were conducted using a microhardness tester (Qness) with a Vickers indenter for 10 s under a 50 g load, by measuring at least 5 measurements and the results were averaged.

A reciprocating wear test was used to evaluate tribological properties of the samples. During sliding tests of the samples, a 5 mm diameter steel ball (100Cr6 steel) (550 HV) with a total sliding distance of 25 m, sliding speed of 3.5 mm/s, load of 3.5 N and a sliding stroke of 5 mm were chosen as wear parameters. The data of the friction coefficient-time curves of the samples were automatically recorded by the computer's own software. After the wear tests, the ball wear volume loss was estimated using the corresponding calculation method below. [16].

$$W=\frac{1}{3}\pi h^2(3R-h)$$

3 Results and discussions

The SEM image taken from the cross-section of the samples is presented in Figure 2. The coating structure of the HDA-1 sample has a uniform appearance in general and does not contain any obvious cracks and gaps (Figure 2a). In addition, there is a sturdy bonding between the coating layer and diffusion zone, and no discontinuity is observed. The thickness of the coating layers belong to the HDA-1 sample was measured as approximately 65 µm for the outer layer and 35 µm for the diffusion layer from the SEM image. As can be interpreted from the SEM-EDS results, the HDA structure consists of an aluminum layer (at%, 99.14 Al, 0.86 Fe) with some intermetallic ($FeAl_3$; at%, 75.19 Al, 24.81 Fe) on the outer surface and a continuous diffusion layer (Fe_2Al_5; at%, 78.37 Al, 21.63 Fe) beneath this layer. While the outer layer is characteristically obtained for HDA process in molten pure aluminum, the formation of the Fe_2Al_5 phase as a diffusion layer is related to the faster growth of this phase owing to its orthorhombic structure in the reaction among molten Al and solid Fe [17][18]. Considering the cross-sectional SEM structure of the HDA-2 sample (Figure 2b), it is seen that the outer layer has a composite appearance composed of aluminum and a high amount of intermetallic, and the thickness of diffusion layer increases approximately 3 times as compared to HDA-1 sample due to applied heat treatment. In addition, there are some Kirkendall pores formed during process attributable to the dissimilar diffusion coefficients of Fe and Al in the HDA-2 sample [19][20]. The formation of intermetallic phases through the diffusion process promoted the strengthening of the bond between aluminum and steel. Although there are some cracks in the HDA-1 sample, it can be said that both coatings have good bonding with the substrate.

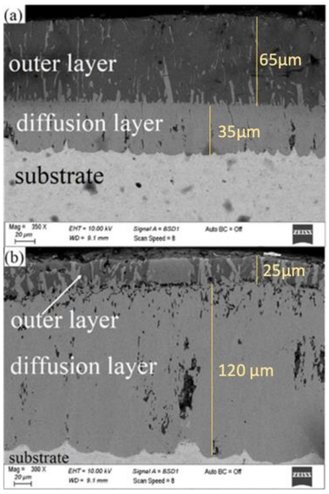

Figure 2. Cross-sectional SEM photographs of the samples: a) HDA-1, (b) HDA-2.

Figure 3 indicates the XRD patterns of HDA-1 and HDA-2 samples. Although both coatings have common phases belong to Al, Fe_2Al_5 and $FeAl_3$, the intensities of these peaks differ. In the HDA-2 sample prepared with additional heat treatment, while the Al peaks showed a dramatic decrease, the $FeAl_3$ phases showed more frequent intensity, and a new $FeAl_2$ phase was formed, as compared to HDA-1 sample.

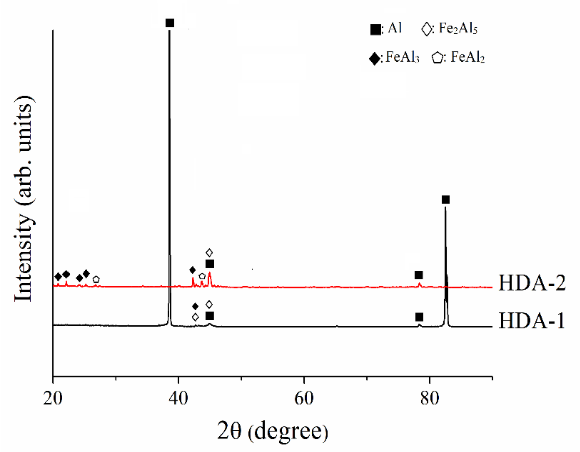

Figure 3. XRD patterns of HDA-1 and HDA-2 samples.

Figure 4 indicates the microhardness values of the both coatings composed of outer layer and diffusion layer. Firstly, when the outer layer hardness values are compared, HDA-2 sample exhibited approximately 2 times higher hardness. This can be attributed to the decrease in the amount of soft aluminum and increase in the amount of intermetallics with high hardness in the outer layer, as confirmed by the XRD result (Figure 3). The increment in hardness can be explained by the intermetallic components $FeAl_2$ and $FeAl_3$, which have a high hardness that can be formed in the relevant system detected in the HDA-2 sample. [21] On the other hand, it is understood that there is no significant difference in terms of the hardness values of the substrate material after the coating processes, and it is compatible with the hardness value of the as-received DP800 material (~264 HV0.05). This indicates that the coating process did not cause any significant changes on the microstructural constituents of the substrate.

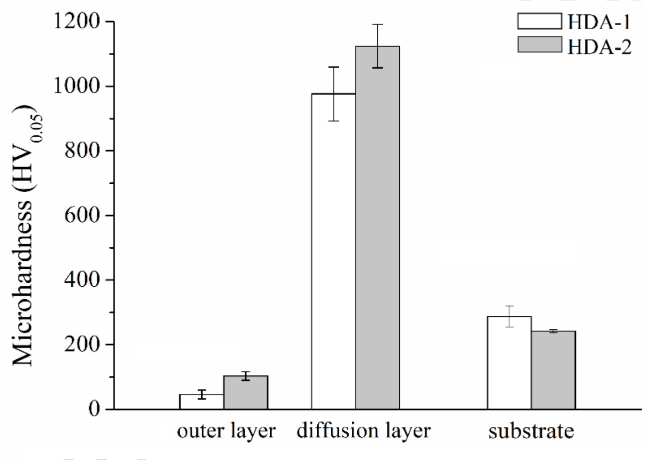

Figure 4. Microhardness values of the coatings.

Figure 5 shows variation of friction curves with time of the as-received, HDA-1 and HDA-2 samples. The friction coefficient of the as-received sample showed a relatively stable change after rising in a short time [22]. In the HDA-1 sample, the friction coefficient showed sudden ups and downs and did not show a stable state. These ups and downs in the friction curve of the HDA-1 sample may indicate that the wear is not uniform on the entire surface. In terms of HDA-2, it is seen that there is no scattering in the friction curve and the friction coefficient is generally stable. The average friction coefficients of the as-received, HDA-1 and HDA-2 samples were measured as 0.71, 0.54 and 0.19, respectively. As the surface hardness increases, it is expected that the wear rate will decrease due to the decrease in the deformation rate due to the decrease in the contact area with the abrasive. The surface with a low coefficient of friction provided a lubricant-like effect, resulting in lower wear-deformation characteristic with steel ball contact. The HDA-2 specimen's relatively high hardness resulted in moderate deformation on tribolayer and consequently, enhanced wear resistance.

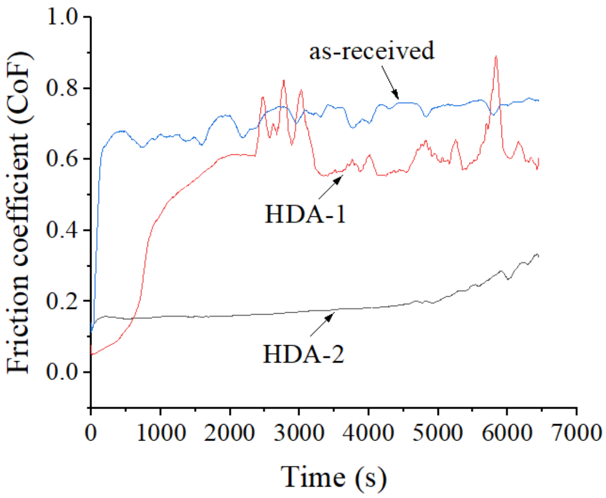

Figure 5. Variation of friction curves with time of the as-received, HDA-1 and HDA-2 samples.

This can be also confirmed from the wear volume loss of the balls used as counter-body during sliding, as shown in Figure 6. Namely, the greatest wear volume loss of the ball was occurred by wear test of HDA-2 sample. Therefore, when the average friction coefficients and wear volume loss of the balls are evaluated together, it can be concluded that HDA-2 is the sample with the best wear resistance. This more protective tribo layer experienced in the wear tests in heat-treated aluminized steel is undoubtedly associated with a thicker hard intermetallic zone. On the other hand, it can be said that HDA-1 sample, which occurred less wear volume loss of ball compared to the as-received sample, has the worst wear property due to lower wear volume ball loss. That is, more wear was observed on the counter material, which was the steel ball in contact rather than the aluminum surface of interest. This inference can be also interpreted that as-received sample has greater hardness than HDA-1 sample, and therefore better wear resistance.

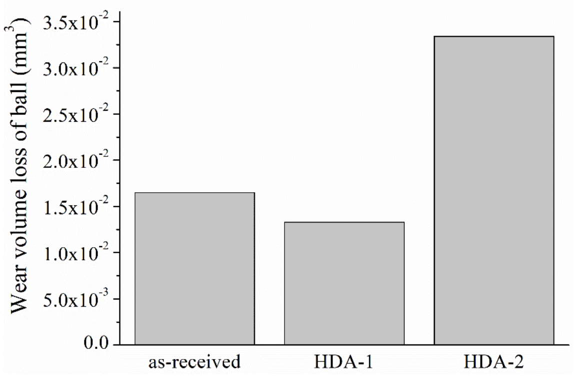

Figure 6. Wear volume loss of ball for the as-received, HDA-1 and HDA-2 samples.

Figure 7 shows worn surface SEM images of the samples after wear tests. In terms of as-received sample (Figure 7a), there is material detachment from the surface, and the main wear mechanism is delamination. The outermost oxide layer, which was fragmented during the wearing process, provided an additional abrasive effect and thus contributed to exhibiting abrasive wear behavior. According to worn surface of HDA-1 sample (Figure 7b), on the other hand, wide grooves are noticeable and abrasive wear scratches and microcracks are prominent, and this demonstrates that the main wear mechanism was abrasive during sliding. The tribolayer of the worn HDA-2 surface (Figure 7c) was enhanced by the formation of more shallow delamination zones with finer wear debris.

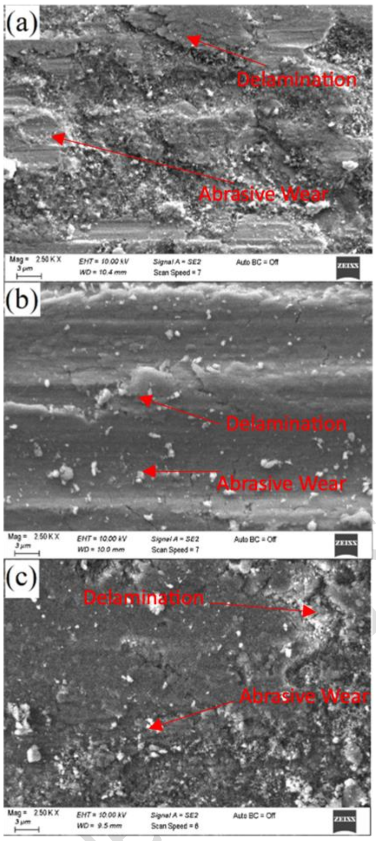

Figure 7. Worn surface of the samples: (a) as-received, (b) HDA-1 and (c) HDA-2 samples.

4 Conclusions

In this study, hot-dip aluminizing (HDA) coatings were successfully produced on the dual-phase (DP800) steel. The influence of heat treatment on the structure of the coating and wear characteristics after HDA process has been thoroughly investigated. On the basis of the experimental findings, the following conclusions can be drawn:

- The HDA structure consists of an aluminum layer with some intermetallic ($FeAl_3$) on the outer surface and a continuous diffusion layer (Fe_2Al_5) beneath this layer. With a heat tretment on HDA coating, the outer layer exhibited a composite appearance composed of aluminum and a high amount of intermetallics ($FeAl_3$, $FeAl_2$), and the thickness of diffusion layer increases approximately 3 times as compared to HDA state.
- The outer layer of heat treated HDA coating exhibited approximately 2 times higher hardness than that of HDA coating.
- In the reciprocating wear test, the HDA sample subjected to heat treatment exhibited the most advanced tribological properties. Although all samples exhibit abrasive wear behavior, delamination areas in the wear direction are less prominent in HDA-2 sample with the highest wearing ball rate.
- The most important result of this study is that post-HDA heat treatment protects DP800 metal surfaces more against wear. This makes DP800 steel, which is widely utilized in the automotive industry, more attractive especially in wear-related applications. Therfore, the investigation of the effect of post-HDA heat treatment on various substrates presents an attractive field for further research on wear-related applications.

5 Author contribution statements

The first author contributed to the experimental processes, their interpretation and publication arrangements. The corresponding author contributed to the creation of the idea, design, literature review, analysis and evaluation of the results.

6 Ethics committee approval and conflict of interest statement

No need for permission from ethics committee for the article prepared. There is no conflict of interest in the article prepared

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