Characterization of PHS coating layer using GDOES technique

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Resumo

O objetivo do estudo foi analisar o perfil de composição química do revestimento Al-Si em aços PHS, por meio da técnica de GDOES. Devido às elevadas temperatura no processo de estampagem a quente ocorre à difusão dos elementos do revestimento e do substrato. Os resultados revelaram que na amostra como recebida, o revestimento apresenta grandes porcentagens de alumínio, silício e oxigênio, que se difundem e diminuem de concentração em direção ao substrato, enquanto que a porcentagem de ferro aumenta. Os resultados das amostras tratadas termicamente em laboratório e do produto final são muito similares, o que indica que o tratamento térmico realizado em laboratório simula corretamente o processo térmico da estampagem industrial. O revestimento é formado por alumínio e ferro, que variam uniformemente até atingir o substrato onde a porcentagem de alumínio diminui em relação à de ferro. A camada revestida também é composta por oxigênio e silício que se difundem em direção ao substrato, onde a porcentagem de oxigênio diminui gradativamente e a de silício não se altera. Os resultados de GDOES mostraram as alterações que ocorrem na camada revestida do aço PHS, e evidenciaram que após o processo de estampagem a quente podem ser formados óxidos e fases compostas por Al-Fe ou Al-Fe-Si.

Abstract

The aim of this study was to analyze the chemical composition profile of the Al-Si coating in PHS steels, using the GDOES technique. Due to the high temperature in the hot stamping process, occurs the diffusion of the coating elements and the substrate. The results showed that in the sample as received, the coating comprises high percentages of aluminum, silicon and oxygen, which diffuse and decrease in concentration towards the substrate, while the percentage of iron increases. The results of the samples heat treated at laboratory and the final product are very similar; indicating that the heat treatment performed at laboratory simulates correctly the heat treatment performed at industry. The coating is formed by aluminum and iron, which vary evenly until reaching the substrate where the percentage of aluminum decreases compared to that of iron. The coated layer also comprises oxygen and silicon that diffuse towards the substrate, however, the percentage of oxygen decreases gradually and the silicon does not change. The results of GDOES showed the changes occurring in the PHS coated layer of Al-Si, and showed that after the hot stamping process, oxides and phases composed of Al-Fe or Al-Fe-Si are formed.

Keywords: PHS, Al-Si, GDOES.

1. Introduction

Press hardened steel (PHS) is a key material to automotive industry, produced by hot stamping process, due to its final properties [1].

The hot stamping consists in to heat up the steel to austenitization temperatures with soak time between 5 and 10 minutes, and then to transfer the steel from the furnace into press tooling where the material is quenched and then get a specific design at the same time [2,3,4].

At the beginning of the process, the steel microstructure comprises ferrite and pearlite, but due to the heat treatment, at the end the microstructure is fully martensitic. Initially the steel shows a tensile strength around 600 MPa which can increase up to 1500 MPa, without springback [2; 3; 4].

The contact with the atmospheric air, during the transfer step in hot stamping, may cause steel oxidation. To avoid this, the steel is protected with metallic coatings [2,3]. The most applied metallic coating in PHS is the aluminum-silicon (Al-Si), which is deposited by hot dip. The bath comprises about 10% of silicon and the remainder content is aluminum. The coating is known to provide a great corrosion resistance by barrier mechanism [3,5].

The combination of complex design, high tensile strength, absence of springback and corrosion resistance, make PHS a key material applied mainly in structural parts of vehicle. As consequence, the vehicle present: increase in safety, lightweight, reduction in fuel consumption and gas emission. These are the targets that the automotive industry must reach [1,2,4].

As a consequence of heat treatment during hot stamping the coating morphology or composition must change due to the diffusion process [3,6]. The present study had the objective of characterizing in terms of depth profiles chemical composition of coating layer and steel substrate, by means of glow discharge optical emission spectroscopy (GDOES), and then to analyze the effect of the heat treatment

2. Experimental

2.1 Materials

The PHS used was the grade 22MnB5 steel, which the chemical composition comprises mainly 0.23% of carbon, 1.18% of manganese and 0.002% of boron. Manganese and boron are known to increase the steel hardenability [3].

Three conditions of samples were tested: the first condition is the material as received, just the material coated without heat treatment or forming. The second condition is the sample heat treated at laboratory following the same parameters of industrial process, in the other words, the sample was heated at 900 °C during 10 minutes, and then cooling down in water. The last condition tested was the commercial product, which was submitted to complete hot stamping process at the industry.

The samples were cut off in dimensions of 20 x 20 mm, and cleaned with ethanol 70%.

2.2 Methods

The distribution of chemical elements between the coating layer and the steel substrate was analysed by means of GDOES technique.

The principle consist in a plasma discharge, the cathodic sputtering is used to remove layer by layer of sample surface. The removed atoms go to the plasma, where they are excited through collisions electrons or Argon metastable atoms. When the excited atoms go back to the ground state, they emit photons with characteristic wavelength to each element [7,8].

3. Results and discussion

The GDOES result for the as received sample is shown in Figure 1

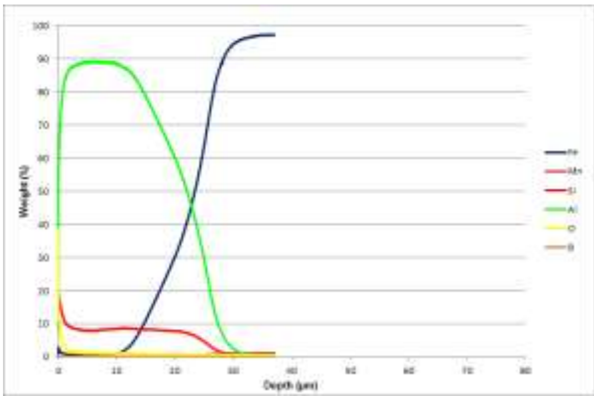

Figure 1- Chemical composition depth profile of as received sample..

According to Figure 1, the coating layer has about 25 µm of thick, from this point it is the steel substrate. At the surface of the coating layer, the chemical composition consists in high amount of aluminum and oxygen (approximately 40% each one), besides that, 20% of silicon. However, the amount of these elements decreases towards the substrate while the amount of iron increases towards the coating layer. At the intersection point the chemical composition is composed by approximately 45% of aluminum and iron and around 10% of silicon. The GDOES result of as received sample is an important tool to do studies related to diffusion, because this result shows the initial state of diffusion (D_0).

Figure 2 shows the depth profile composition of heat treated sample carried out at laboratory.

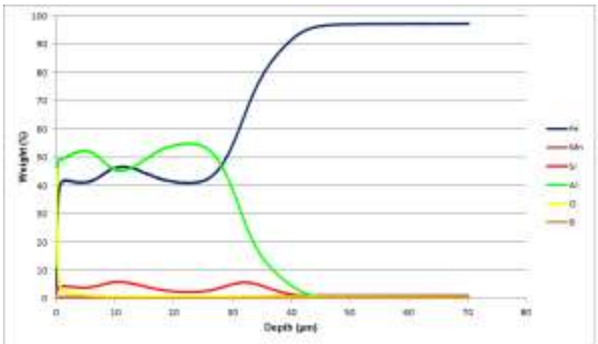

Figure 2- Depth profile composition result for the heat treated sample at laboratory.

It is possible to see that the profile is completely different from Figure 1, a consequence of the high temperature of the process that promotes the diffusion of the elements. The coating layer has around 30 µm of thick. Just at the surface the presence of oxygen is predominant; however, its amount decreases to less than 5%. Besides the oxygen, the coating layer consists in aluminum, iron and silicon. The amount of aluminum and iron varies simultaneously in whole coating layer. From the depth around 30 µm the amount of iron increases and the aluminum decreases at the steel substrate. The amount of silicon varies slightly in the coating layer, moreover it is seen that when the amount of aluminum is high, the amount of silicon is low. The GDOES results suggests that the coating layer may comprises phases of Al-Fe or Al-Fe-Si, which is indicated in literature and can be observed by other techniques, such as, X-rays diffraction or scanning electron microscope [5,9].

The depth profile composition of the sample from the final product, that means, from the industry, is shown in Figure 3.

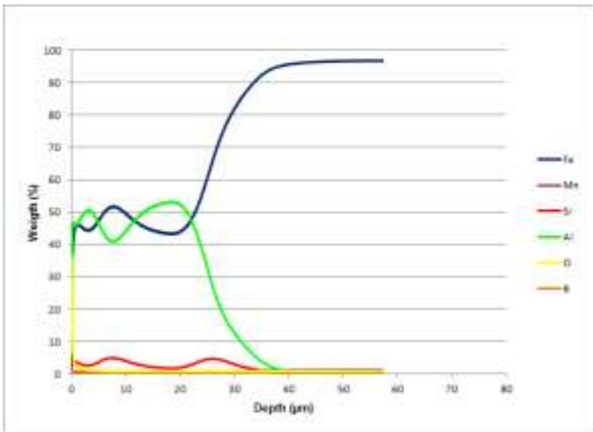

Figure 3- Depth profile composition result for the commercial product.

Slight differences are observed from Figure 2 to Figure 3, which is an indication that the heat treatment performed at laboratory, following the industrial conditions of temperatures and time, simulates correctly the heat treatment performed at industry.

4. Conclusion

The GDOES technique is an important tool to analyse the chemical composition in function of the depth. Moreover, it is a helpful technique in diffusion studies, once it offers an experimental result, as base to a simulated one.

Related to the Al-Si coating, it was seen the consequence of the heating in the hot stamping process, which resulted in the chemical elements diffusion and completely change on the depth profile. From the GDOES, it is suggested that the coating layer comprises phases of Al-Fe or Al-Fe-Si, and oxides.

5. References

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