

The use of X-ray diffraction for phase identification of press hardened steels

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Abstract

The numbers of hot stamped components have been steadily increasing in the automotive industry. Press hardened steels are generally used in hot stamping process since at the end of the process, the steel may achieve a tensile strength about 1500 MPa. The hot stamping process consists to heat the steel blank at total austenitization temperature and to transfer the blank into the press tooling for shaping and fast cooling to fully martensitic transformation. The transfer of the blank from the furnace to the press might promote at some extent, steel oxidation. The application of metallic coatings avoids this hindrance. The Al-Si has been the most applied coating on steel. In parallel, alternative coatings such as Zn-Ni have been developed. It is known that the heating causes chemical elements diffusion, which results in intermetallic formation between the elements presents in the coating and in the substrate. This study had the objective of characterizing hot stamped and coated 25MnB5 steel samples with Al-Si and Zn-Ni, with X rays diffraction (XRD) technique. Some literature suggested phases such as Zn-Fe, Zn-Ni-Fe or Al-Fe-Si were not seen on XRD results. Nonetheless, X-ray diffraction detected the presence of ZnO and α -Fe for Zn-Ni coated steel plate and Al_5Fe_2 , $AlFe_3$ and α -Fe for the Al-Si coated steel plate.

Key words: PHS, phase, X-ray diffraction.

Resumo

É crescente o número de componentes estampados a quente destinados a indústria automotiva. Os aços endurecidos por estampagem (PHS) são geralmente usados no processo de estampagem a quente porque ao final do processo o aço atinge resistência mecânica próxima a 1500 MPa. O processo de estampagem a quente consiste em aquecer um blank em temperaturas de total austenitização e transferi-lo do forno de aquecimento para uma prensa. Nesta prensa, ocorre a conformação mecânica e o resfriamento brusco para total transformação martensítica. O estágio de transferência do forno para a prensa pode promover alguma oxidação do aço. A aplicação de revestimentos ao blank evita esse fenômeno. O revestimento Al-Si tem sido o mais aplicado. Paralelamente, estudos de revestimentos alternativos como o Zn-Ni estão sendo realizados. Sabe-se que o aquecimento favorece a difusão dos elementos químicos que resulta na formação de intermetálicos entre os elementos presentes no revestimento e no substrato. Este estudo teve o objetivo de caracterizar as fases presentes em amostras estampadas e revestidas por Zn-Ni e Al-Si pela da técnica de difração de raios X. Não foram identificadas fases sugeridas pela literatura como Zn-Fe, Zn-Ni-Fe ou Al-Si-Fe. Mesmo assim a difração de raios X detectou a presença de ZnO e α -Fe para a chapa de aço revestida com Zn-Ni e para a chapa de aço revestida com Al-Si, a presença de Al_5Fe_2 , $AlFe_3$ e α -Fe.

Palavras chaves: PHS, fases, difração de raios X.

1. Introduction

The application of hot stamped components in automotive industry is steadily increasing. It is a strategic product to help the automakers to achieve the INOVAR - AUTO targets regarding fuel consumption and emissions. The aim is to produce lighter and safer vehicles, and then reduces the CO₂ emissions [1].

Press hardened steels (PHS) are boron-manganese steels. They are usually used in hot stamping process achieving after processing a tensile strength around 1500 MPa. Moreover, the spring back effect is not seen in the shaping process as a consequence of steel chemical composition combined with high temperatures [2]; [3] [4].

The hot stamping process consists in heating a steel blank at total austenitization temperatures and to transfer the blank into the press tooling for forming and fast cooling to fully martensitic transformation, as shown in Fig. 1. At the beginning of the process the steel has around 600 MPa of tensile strength; at the end it increases to 1500 MPa. The blank transfer from the furnace to the press might promote some steel oxidation. In order to avoid this phenomenon, coatings are applied on the blank [5]; [6].

The Al-Si coating has been the most applied on PHS by hot dip. The bath consists in 10 % in mass of silicon in aluminum. However, it is a patented material developed by ArcelorMittal known as USIBOR. Thus, alternative coatings zinc base, have been developed. Zn-Ni is one of alternative to Al-Si; it is an electrodeposited coating, which consists in around 15 % in mass of nickel [5]; [6]. It is known that the heating causes chemical elements diffusion that results in intermetallics formation amongst the elements presents in the coating and base metal [5].

The present study had the objective of characterizing the formed phases in samples coated with Al-Si and Zn-Ni after hot stamping, through X-ray diffraction technique (XRD).

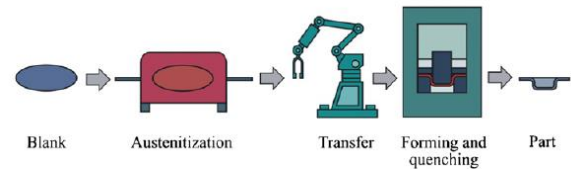


Figure 1. Schematic drawing of the hot stamping process [6].

2. Experimental

2.1 Materials

The samples were removed from the B - pillar inner, from a commercial and experimental parts of the 22Mn5B steel plate 1.2 mm thick. Two series of samples were taken: Sample I: Zn-Ni coated (20x20 mm²). Sample II: Al-Si coated (20x20 mm²). Due to the shape of the part, it was not possible to obtain totally flat samples.

2.2 Methods

X-ray diffraction was undertaken at Bragg-Brentano geometry, with Cu anode. The angle of scanning was (2 θ): 4° - 70°.

3. Results and discussion

Fig. 2 shows the result of X ray diffraction pattern for sample I, Zn-Ni coated.

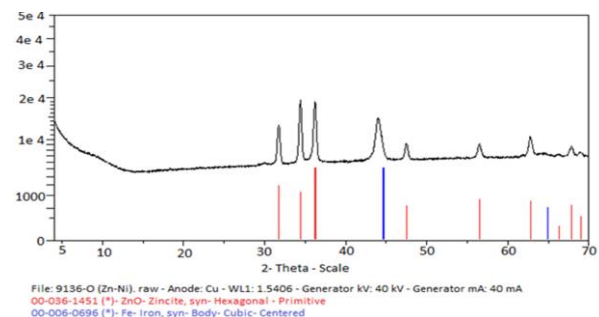


Figure 2. X-ray diffraction pattern of Zn-Ni coated plate showing the presence of ZnO and α -Fe.

The diffraction pattern of Zn-Ni coating only shows the presence of ZnO and α -Fe. This result might not show all the present phases because the sample was not totally flat, as it is required according to Bragg's law. An important observation is the absence of phases

composed by Zn-Ni or Zn-Fe-Ni. Kondratiuk et al showed in their study the presence of γ -(Ni₅Zn₂₁) intermetallic, however this phase was not observed in the present study [5].

Fig. 3 shows the result of X-ray diffraction pattern for sample II, Al-Si coating.

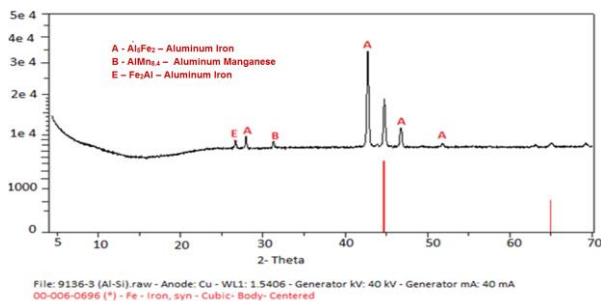


Figure 1. X-ray diffraction pattern of Al-Si coating showing the presence of Al₅Fe₂ (peak A) AlFe₃ (peak E) and α -Fe.

The corresponding diffractogram for sample II shows the presence of Al₅Fe₂ (peaks A) AlFe₃ (peak E) and α -Fe. Al₅Fe₂ is a brittle phase and it may promote cracks in coating layer. On the other hand, AlFe₃ improve the corrosion resistance and it shows higher ductility in comparison with Al₅Fe₂ [7]. The condition of sample might imply in the absence of phases composed by Al-Fe-Si. As reported by Windmann et al the silicon occupies a vacancy in Al₅Fe₂ phase, or it may increase the amount of phase Al₈Fe₂Si [6]. The phase AlMn_{8.4} (peak B) was identified, but information about this phase was not found in literature.

4. Conclusion

In respect to sample I, Zn-Ni coated steel plate, ZnO was observed by X-ray diffraction as expected. However, phases composed by Zn-Fe or Zn-Fe-Ni were not identified.

The corresponding X-ray diffraction pattern of sample II, Al-Si coated steel plate, shows the phases Al₅Fe₂, AlFe₃ and AlMn_{8.4}, but the reported phases composed by Al-Fe-Si were not identified.

Even so X-rays diffraction is an important technique for characterization of crystalline materials, mainly during product development.

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