INFLUENCE OF HEATING RATE ON MICROSTRUCTURE OF GALVANNEALED COATING DURING HOT STAMPING*

Henrique Lacerda Eleutério[1]
Aldo Henrique de Almeida Barbosa[2]
Vicente Tadeu Lopes Buono[3]

Abstract
The automotive industry's interest in galvannealed (GA) boron steel for hot forming processes application has been steadily increasing due to its excellent surface quality, galvanic protection provided to the parts, good paint adhesion and better weldability in relation to other coatings for this application. In this study, samples of 22MnB5 galvannealed steel were heat treated and hot stamped on a water cooled die in laboratory scale. The influence of heat treatment on stability of coating and steel was assessed by using initial heating rates 11.0ºC/s and 15.0ºC/s.
Microstructural evolution of galvannealed coating and structural integrity of steel during hot forming were investigated by scanning electron microscopy with energy dispersive spectroscopy analysis (SEM/EDS/Line scan), X-ray diffraction (XRD) and optical microscopy (OM). It was possible to quantify the Fe-Zn phase evolution during hot forming, to understand the effect of these heating rates on the product properties and to establish an optimized heat treatment for hot forming of these steel. The heat treatment with the heating rate of 15.0ºC/s presented greater operational flexibility and final products free from surface defects or cracks in the substrate.
Keywords: GA coating; 22MnB5 steel; Press hardening steel; Hot stamping.

[1] *Metallurgical Engineer, Master of Science in Metallurgy and Materials, Researcher Engineer at Research and Development of Process Management, Usiminas, Ipatinga, MG, Brazil.*
[2] *Metallurgical Engineer, Doctor of Engineering in Metallurgy and Materials, Manager at Research and Development of Products Management, Usiminas, Ipatinga, MG, Brazil.*
[3] *Physicist, Doctor of Engineering in Metallurgy and Materials, Professor at Metallurgical Engineering and Materials Department of Federal University of Minas Gerais (UFMG), Belo Horizonte, MG, Brazil.*

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1 INTRODUCTION

In the last years, the automotive industry interest for greater market competitiveness has promoted the development of new steels grades that encourage the desired weight reduction, responsible for the higher fuel economy and reduction of the environmental impacts, and are also able to guarantee the safety of the user without increasing the cost of production. The more stringent requirements related to the performance of a vehicle during a collision can be satisfied by a thicker cold stamped steel. However, the use of a thicker cold stamped steel increases the overall weight of the vehicle. Also, these steels have limited formability with tensile strength (TS) up to 1200 MPa and sharp springback effect. The forming of complex parts (such as B-pillar) requires processes such as hot stamping, which allow the production of pieces with higher geometric complexity and minimizing geometric defects caused by the springback effect.

Hot stamped 22MnB5 steel with Galvannealed (GA) coating has been gaining ground every year in the automotive industry due to its excellent surface quality, the galvanic protection it provides to the parts, good weldability and adhesion of the paint layer to the substrate at the end of the process relative to other coatings for hot stamping application. Also, there is a high expectation of consumption increasing of 22MnB5-GA steel in the coming years due to its excellent characteristics [1]. This steel has high mechanical resistance at the end of the hot stamping coupled with excellent ductility during forming. The microstructure of this steel before the hot stamping is composed of ferrite-perlite, and its TS is 600 MPa, which has good ductility. After the hot stamping process, the microstructure is predominantly martensitic and the TS significantly increases, obtaining values more than 1500 MPa [2].

However, the main challenge faced by Usiminas in the development of this new product was to ensure the structural integrity of the coating layer containing Zn and avoid the liquid metal embrittlement (LME) during the hot forming process. So, it is necessary to ensure that the coating layer containing Zn does not vaporize during the austenitization treatment since the temperatures involved in the heat treatment are higher than the Zn melting temperature.

Therefore, it becomes necessary to understand and determine the transformation of the coating in the hot stamping process of 22MnB5-GA steel.

In this study, the influence of the heating rates on microstructural evolution of GA coating and structural integrity of steel were investigated in laboratory scale during the hot stamping process.

2 MATERIAL AND METHODS

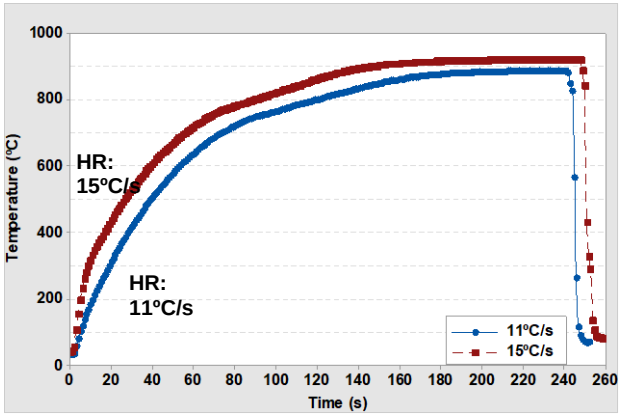
The samples used in this study were an industrially processed 22MnB5-GA steel with a thickness of 1.80 mm, provided by Unigal Usiminas. The typical chemical composition of 22MnB5-GA steel is shown in table 1. The samples were processed in a Continuous Galvanizing Line (CGL) using a galvannealing treatment with high inductive heating temperature (IHT) and soaking for up to 35 s.

Table 1. Chemical composition of investigated 22MnB5-GA steel (%wt.)

C	Si	Mn	P	Al	B	Ti
0,22 a 0,27	≤0,5	1,10 a 1,50	≤0,03	≥0,01	0,0005 a 0,004	0,020 a 0,055

The samples were heated in an electric furnace with a non-inert atmosphere and heating rates for up to 35 s of 11.0ºC/s and 15.0ºC/s for the evaluation of the hot stamping parameters in laboratory scale. The control of the initial heating rate for up to 35 s is important to ensure that the Zn

rich phases do not evaporate in the first sections of heat treatment. The specimens were quenched in water after the required dwell time was reached to evaluated the microstructure and understand the interaction of coating and steel substrate. Figure 1a shows the temperature profiles of the samples during the heat treatment at the electric furnace. The samples were formed in a laboratory scale hydraulic press of 40 t with a water cooled die after the heat treatment. The arrangement of the equipment is shown in figure 1b.



a) Temperature profile

b) Equipment arrangement

Figure 1. a) Temperature profiles of the samples during the heat treatment and b) equipment arrangement used in the hot stamping simulation.

The chemical composition and phase determination of the GA coating before the hot forming process were investigated by inductively coupled plasma optical emission spectrometry (ICP-OES) and X-ray diffraction (XRD). The surface morphology, microstructure and chemical composition analysis of the coating, before and after hot forming, were investigated by scanning electron microscopy with coupled energy dispersive X-ray spectroscopy (SEM/EDS/Line scan). The values of the Line scan profile acquired in the cross-section of the coating were interpolated in the Fe-Zn phase diagram shown in figure 2 to verify the present phases in the referred coating region. The intersection of the Zn and Fe curves initially represents the coating/substrate interface. Phase determination was performed considering that the ζ (zeta) phase contains 5% to 6% Fe; δ (delta), 7% to 12% Fe; Γ1 (gamma 1), 17% to 19% Fe, and Γ (gamma), 23% to 28% Fe [1,3,4].

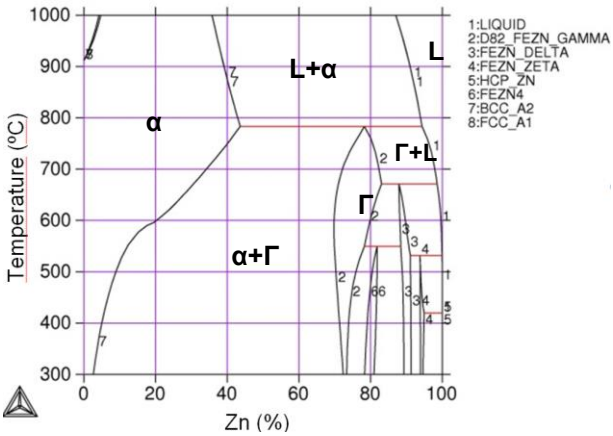


Figure 2. Fe-Zn diagram obtained by Thermocalc® analysis.

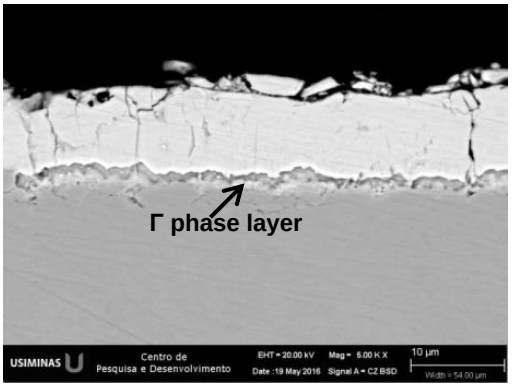
The microstructure of the bending region (region exposed to high stress forces) and stretching region (region exposed to friction forces) of the pieces after the hot forming was evaluated by optical microscopy (OM) and Zn mapping analysis by SEM/EDS. The grain boundaries embrittlement by liquid Zn penetration (LME) in these regions was assessed. According to Drillet *et al.* [5], the bending areas are subject to macrocracks (≥100 μm) formed by LME and the stretching regions are subject only to microcracks, formed by the friction between the pieces and die. The mechanical properties of the substrate were evaluated by tensile testing using

specimens in agreement to ASTM A370 standard [6].

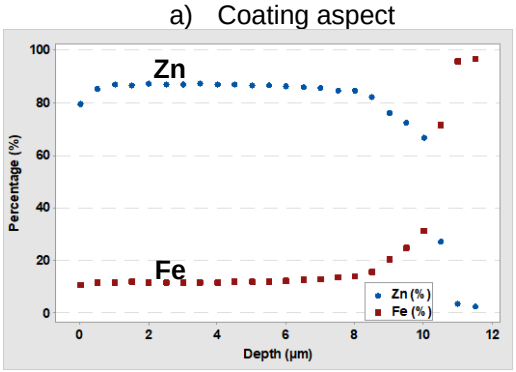
3 RESULTS AND DISCUSSION

3.1 Aspect of the GA Coating Before to the Hot Stamping Process

The aspect of the GA coating before to the hot forming process is shown in figure 3. This GA coating produced with a high TIH presented higher Fe content and thicker Γ layer than typical GA coatings intended for cold forming [3]. The higher Fe content and Γ layer thickness indicate that this coating exhibit suitable characteristics to the hot forming process. These characteristics indicate that this GA coating is less susceptible to mass loss by Zn vaporizing at the beginning of the heat treatment [1].



a) Coating aspect



b) SEM/Line scan profile acquired in the coating cross section

Figure 3. SEM/EDS images and chemical composition in the GA coating.

Figure 4 shows the XRD diffractogram of the GA coating. Its microstructure is constituted by Γ, δ and ζ phase layers. The presence of ζ phase in less extent than δ and Γ phases suggests that this coating is less susceptible to form liquid phase in the beginning of the heat treatment [1].

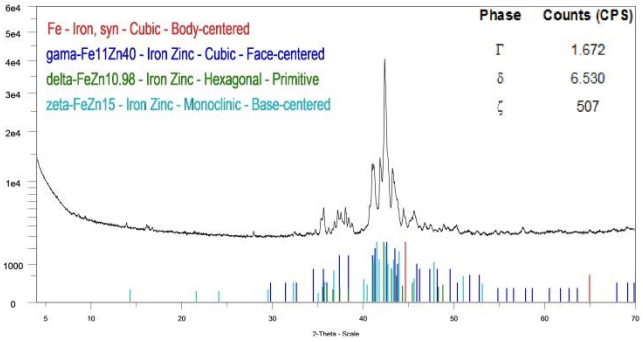


Figure 4. XRD diffractogram of the GA coating before the hot stamping process.

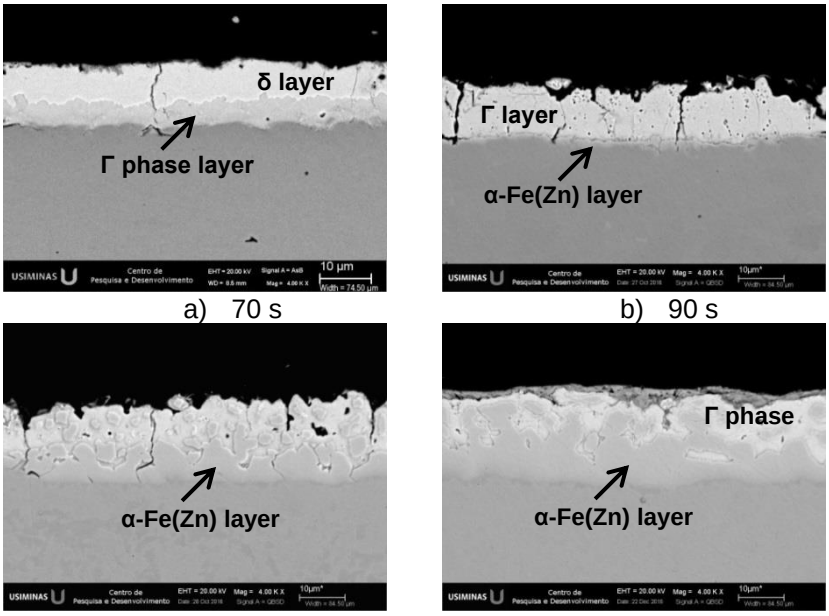
3.2 Coating Transformation During the Hot Stamping Process

Figures 5 to 8 show the results of the microstructure evolution analyses of the coating on 22MnB5 steel during the heat treatment. It is noticed the coating transformation occurs more intensively in the heat treatment using the initial heating rate of 15.0°C/s. For this condition, figure 7, the growth of the Γ (30% Zn-Fe) phase layer occurs so intensively at time 70 s. According to these results, it is evident the stepwise and homogeneously occurrence of the transformation of the Fe-Zn phase crystals present in the GA coating, mainly in the formation and distribution of the new phases formed in the coating. Among these newly formed phases, it is observed the formation of well-defined layer limits. The formation of new Γ crystals was provided from the transformation of the ζ and δ by their Fe enrichment.

Following the heat treatment, a ferritic solid solution layer (α-Fe(Zn)) is observed at coating/substrate interface at 90 s. The formation of this layer is favored by the continuity of Fe diffusion into the coating, figures 5b, 6b, 7b and 8b.

The evolution of the α-Fe(Zn) (70%Fe-Zn) layer occurs through two distinct growth mode. Firstly, it is noted the growth of the continuous α-Fe(Zn) layer formed on

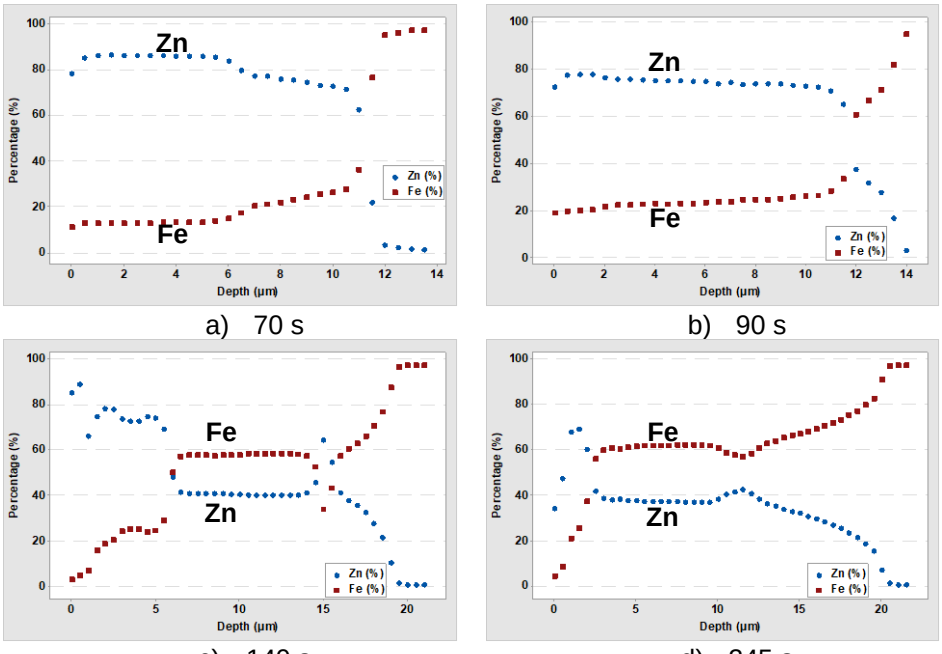
coating/substrate interface. The second mode is the formation and growth of the α-Fe(Zn) globular islands in different regions of the coating. These two types of α-Fe(Zn) layer formation and growth are observed at time 140 s, figures 5c, 6c, 7c and 8c.



a) 70 s b) 90 s

c) 140 s d) 245 s

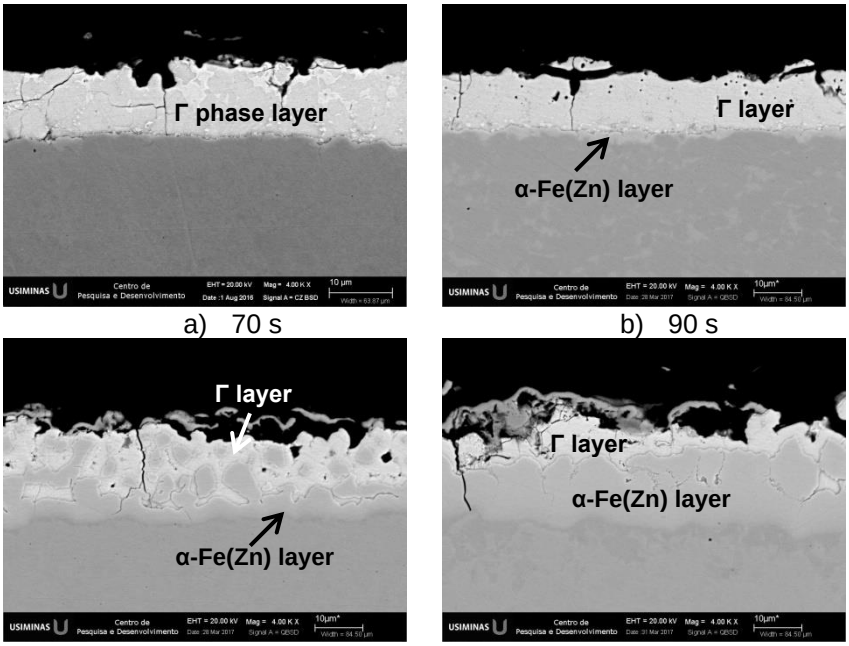
Figure 5. SEM images of coating evolution during the heat treatment with the heating rate of 11.0ºC/s.



a) 70 s b) 90 s

c) 140 s d) 245 s

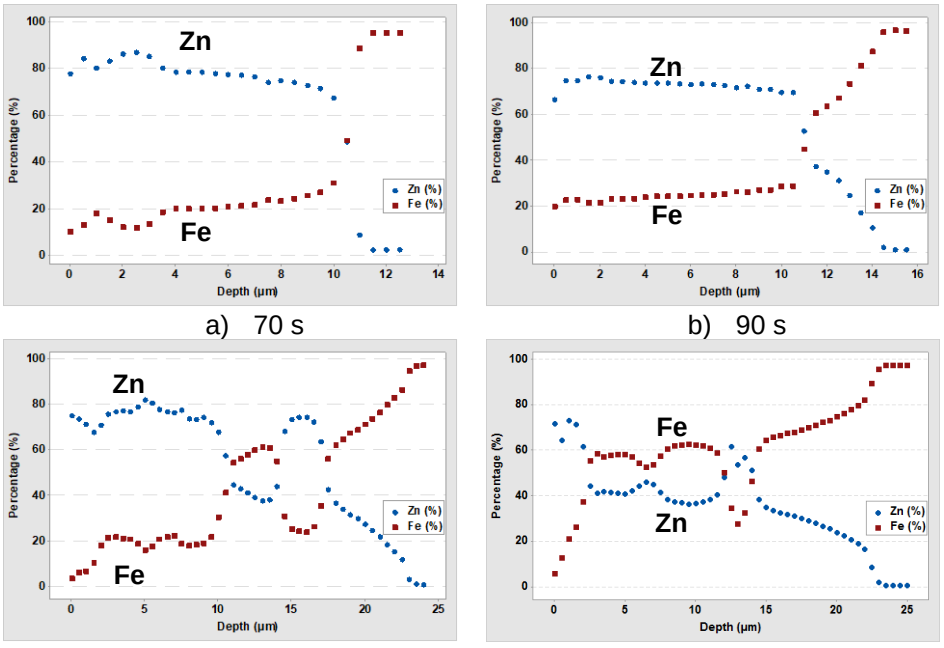
Figure 6. Chemical composition profile of the coating cross section obtained by SEM/EDS/Line scan during the heat treatment with the heating rate of 11.0ºC/s.



a) 70 s b) 90 s

c) 140 s d) 245 s

Figure 7. SEM images of coating evolution during the heat treatment with the heating rate of 15.0ºC/s.



a) 70 s b) 90 s

c) 140 s d) 245 s

Figure 8. Chemical composition profile of the coating cross section obtained by SEM/EDS/Line scan during the heat treatment with the heating rate of 15.0ºC/s.

Despite the use of two different thermal parameters, the formation of the α-Fe(Zn) layer occurs at 90s of heat treatment for both situations. However, the growth of α-Fe(Zn) occurs more intensively for the material heat treated with the heating rate of 15°C/s, representing 40% of the coating layer in this time.

Figure 9 shows the evolution of α-Fe(Zn) layer calculated by image analysis. The evolution of the α-Fe(Zn) layer during the heat treatment is higher for the process

with high initial heating rate than the low heating rate.

The formation and growth of this α-Fe(Zn) layer is of great importance for the hot stamping process, because it acts as a barrier and prevents the penetration of Zn liquid in the grains boundaries and cracks propagation in the substrate.

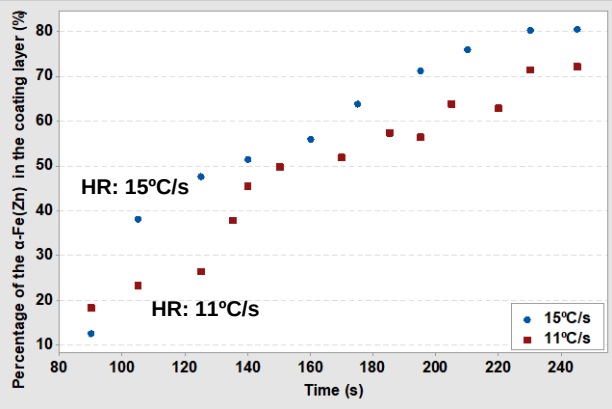


Figure 9. Evolution of the α-Fe(Zn) layer during the heat treatments calculated by image processing analysis.

From this time up to 245 s, the morphology, microstructure and chemical composition of the coating completely change and become consisted of two distinct layers as shown in figures 5d and 7d. The first layer is majority and constituted by the solid solution α-Fe(Zn) and the second one is formed in the coating surface and constituted by the intermetallic Γ phase. In addition, above the surface is evidenced the formation of a thick oxide layer. This oxide layer is constituted by oxides of Mn, Si, Al and Zn. The formation and growth of this oxide layer have great importance for the hot stamping mainly protects the coating against loss of mass, ensuring its integrity when it is subjected to elevated temperatures [7,8].

3.3 Characteristics of the Coating after the Hot Stamping

Figure 10 shows the aspect of the samples hot formed in the pilot press.

Figure 10. Aspect of the samples after hot stamping.

Tables 2 and 3 show the values of YS and TS of the samples submitted to the heat treatments with different heating rates are similar after the hot forming. According to these results, it is observed for the samples submitted to the heat treatment with an initial heating rate of 11.0ºC/s, times higher than 150 s do not affect and produce additional gains to heat treatment. For samples submitted to the heat treatment with an initial heating rate of 15.0ºC/s, times from 120 s also do not produce additional gains.

Table 2. Values of the mechanical of the samples heat treated with initial heating rate of 11.0ºC/s

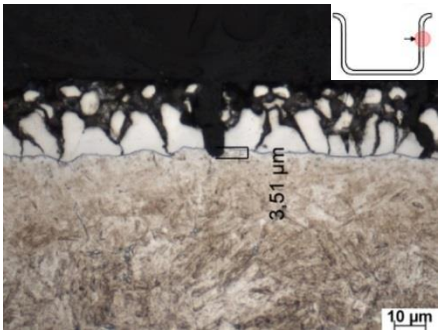
Time (s)	YS (MPa)	TS (MPa)	Uniform Elongation (%)	Total Elongation (%)
70	452	618	11.3	21.6
90	526	710	7.9	14.1
150	1531	1781	2.5	2.9
245	1533	1764	2.1	2.6

Table 3. Values of the mechanical properties of the samples heat treated with initial heating rate of 15.0ºC/s

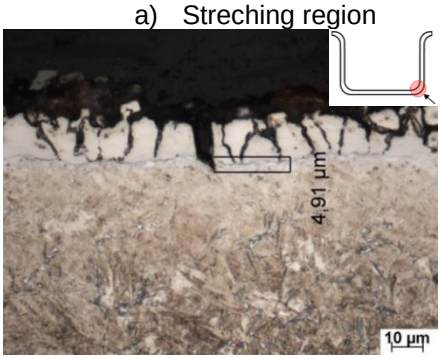
Time (s)	YS (MPa)	TS (MPa)	Uniform Elongation (%)	Total Elongation (%)
70	465	624	11.8	22.7
90	486	866	10.7	14.5
120	1397	1789	4.1	6.7
245	1361	1729	3.6	6.2

The microstructure of the substrate heat treat in both conditions is predominantly composed by martensite, evidencing the effectiveness of the heat treatment in the

laboratory scale and its similarities with the industrial process, figures 11 and 12 [1].
The stamped samples also were evaluated according to presence of intergranular cracks and originated cracks by the liquid metal penetration in the grain boundaries (LME). The results of the metallographic analyses of the bending region, a critical condition where the grain boundaries are subjected to tensile stress, and of stretching regions of the stamped parts are shown in figures 11 and 12. These results show the presence of cracks in the bending and stretching regions of the parts obtained with heating rate of 11.0ºC/s. The cracks present size between 3.0 µm and 5.0 µm. It is observed that the presence of this crack is associated with the embrittlement due to the penetration of liquid Zn in the grain boundaries of this region (LME), figure 13 [8]. The growth of the solid solution layer for this condition is lower than the heating rate of 15.0°C/s. The coatings heat treated with a heating rate of 11.0°C/s remains for a longer time susceptible to penetration of liquid Zn in the grain boundaries during the heat treatment.

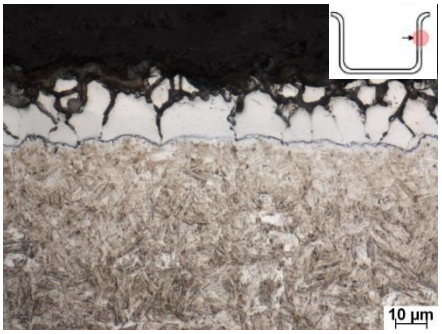


a) Streching region

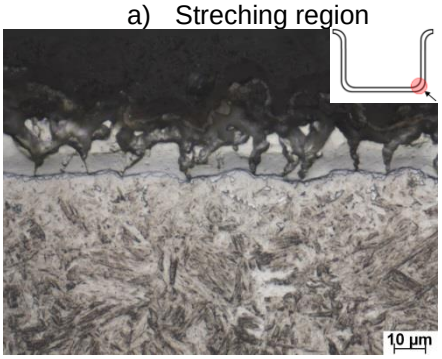


b) Bending region

Figure 11. OM images of microstructure of the samples heat treated with an initial heating rate of 11.0ºC/s.

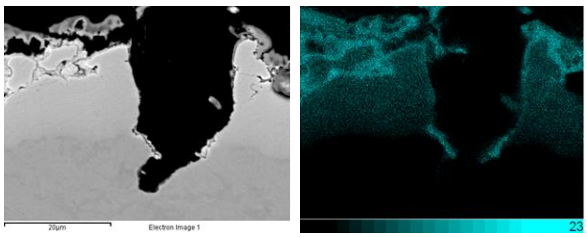


a) Streching region

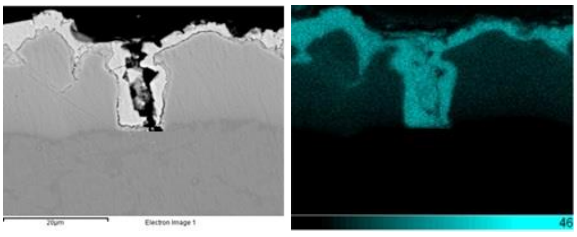


b) Bending region

Figure 12. OM images of microstructure of the samples heat treated with an initial heating rate of 15.0ºC/s.



a) Initial heating rate of 11.0ºC/s



b) Initial heating rate of 15.0ºC/s

Figure 13. Mapping analyzes of the Zn element acquired by SEM/EDS in the coating and substrate of samples heat treated with different heating rates.

4 CONCLUSION

The heat treated 22MnB5-GA steel with heating rate for up to 35 s of 15.0ºC/s in laboratory scale produced a coating suitable to withstand the hot forming application. The parts processed in this condition presented excellent surface quality and structural integrity. The microstructure of the substrate was predominantly martensitic and the mechanical properties (YS and TS) of the samples was as desired for hot-stamped parts. It was observed the presence of cracks associated to LME in the parts obtained with initial heating rate of 11.0ºC/s.

The coating obtained after the both heat treatment consisted of two distinct layers. The first layer consists of the α-Fe(Zn) solid solution (70% Fe-Zn). The second layer is above the first one and consists of the intermetallic Γ phase (70% Zn-Fe). This α-Fe(Zn) layer is of great importance for the process, as it acts as a barrier protecting the substrate against the penetration of liquid Zn in the grain boundaries and the cracks propagation.

It is observed for the samples submitted to the heat treatment with a heating rate of 11.0ºC/s, times higher than 150 s do not affect and not produce additional gains to heat treatment. For samples submitted to the heat treatment with a heating rate of 15.0ºC/s, times over 120 s also do not produce additional gains. These results suggest possibilities of productivity gains in industrial hot stamping lines with the use of shorter processing times of the material.

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