Study of cracks propagation inside the steel on press hardened steel zinc based coatings

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Al-Si coating is the most suitable solution for main Hot-Forming applications, particularly in terms of process window for the hot stampers and in perforating corrosion resistance after austenitization. But for some specific cases, a few customers require galvanic edge protection. So, in order to satisfy this requirement zinc based coatings were developed for Hot-Forming.

On these Zn based coatings a full microstructural characterization was carried out on the coating influence on the steel/coating interface during the hot stamping. It appears that some cracks propagation is always observed inside the steel with Zn based coatings. Two separate cases corresponding to two mechanisms have to be distinguished: Macro and Micro-cracks. The MACRO-cracks propagation is related to a liquid zinc penetration inside the previous austenitic steel grains boundaries. This is encountered for areas showing a high level of tensile stress with remaining liquid Fe-Zn phases in the coating during the deformation. Thus, a cold deformation is a preliminary step for GI coating. The MICRO-cracks propagation is related to a friction issue between the coating surface and the tools at high temperature. The higher micro-cracks density is consequently observed on areas more sensitive to friction. The phases inside the steel responsible for this propagation have been identified. Some solutions to avoid these phenomena are proposed, particularly in the case of the micro-cracks for Direct Hot-Forming applications (GA coatings).

Keywords:
PHS Zinc coating, Cracks propagation, Hot-Forming, boron steels, 22MnB5 AlSi

INTRODUCTION

In the design of new body-in-white structures, the hot stamping process appears to be the most important technology to produce complex parts of advanced high strength steels, particularly well adapted for higher passive safety and weight reduction which are the main objectives of the automotive industry. ArcelorMittal has developed an aluminized boron steel (22MnB5 AlSi), for direct hot stamping applications [1]. This pre-coated solution has been proposed to prevent scaling and decarburization during the austenitization step. Moreover the AlSi coating gives a very good protection against corrosion, particularly in terms of perforating corrosion.

In the direct hot stamping process, the steel blank is heated in a furnace for a few minutes (3-10 min) to a temperature range of 880-930°C in order to get a steel fully austenitic; then it is quickly transferred (5-7 s) to a press in which it is immediately formed into the desired component shape and simultaneously hardened by die quenching (cooling rate higher than 50°C/s to avoid the bainitic transformation in the deformed areas). This direct hot stamping process is described in figure 1. The final automotive part has then a fully martensitic microstructure and shows a Tensile Strength value around 1500 MPa.

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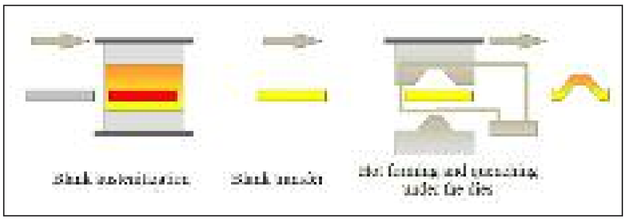


FIG. 1 ***Hot stamping process.***
Processo di stampaggio a caldo.

In order to answer to customers who desire an additional cathodic protection, ArcelorMittal has developed zinc pre-coated steels for hot stamping: the galvanized Zn 22MnB5 GI which is dedicated to the indirect hot stamping process (95% of cold deformation followed by the austenitization step + 5% of hot deformation to complete the calibration), and the galvannealed Zn-Fe10% 22MnB5 GA which can be processed according to direct or indirect hot stamping. The coatings thicknesses after heat treatment are in the range 19-26 μm. In that case, it is necessary to adapt the austenitization temperature to be systematically lower than 905°C in order to avoid Zn coating burning inside the oven.

This study is focused on the understanding of the mechanisms of macro and micro-cracks propagation inside the steel during the Hot Forming step.

Zn base coatings developed by ArcelorMittal: 22MnB5 GI and 22MnB5 GA

The Zn-based coatings are produced by hot-dipping the steel into

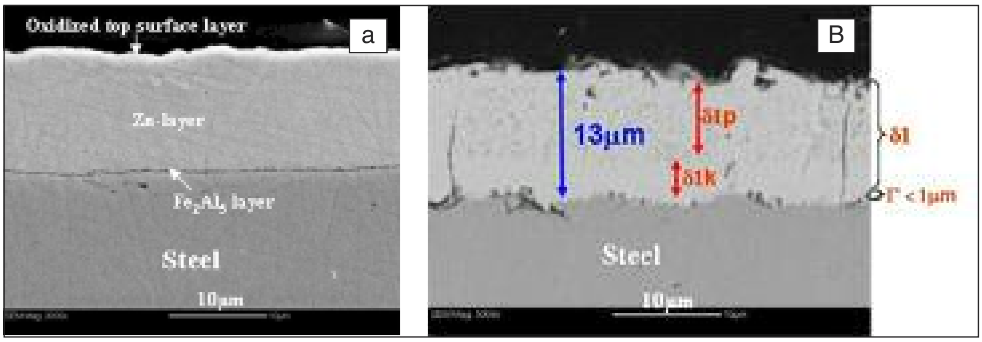


FIG. 2
a) GI-coating microstructure obtained after hot dipping, (b) GA-coating microstructure obtained after hot dipping and diffusion annealing.

(a) Microstruttura del rivestimento GI ottenuta dopo zincatura ad immersione a caldo, (b) Microstruttura del rivestimento GA ottenuta dopo zincatura ad immersione a caldo e ricottura di diffusione.

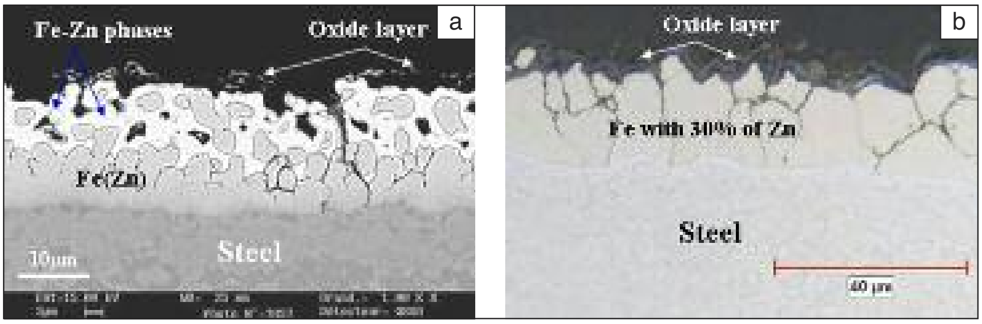


FIG. 3
(a) GI-coating microstructure obtained after hot stamping, (b) GA-coating microstructure obtained after hot stamping.

*(a) Microstruttura del rivestimento GI ottenuta dopo stampaggio a caldo,
(b) Microstruttura del rivestimento GA ottenuta dopo stampaggio a caldo.*

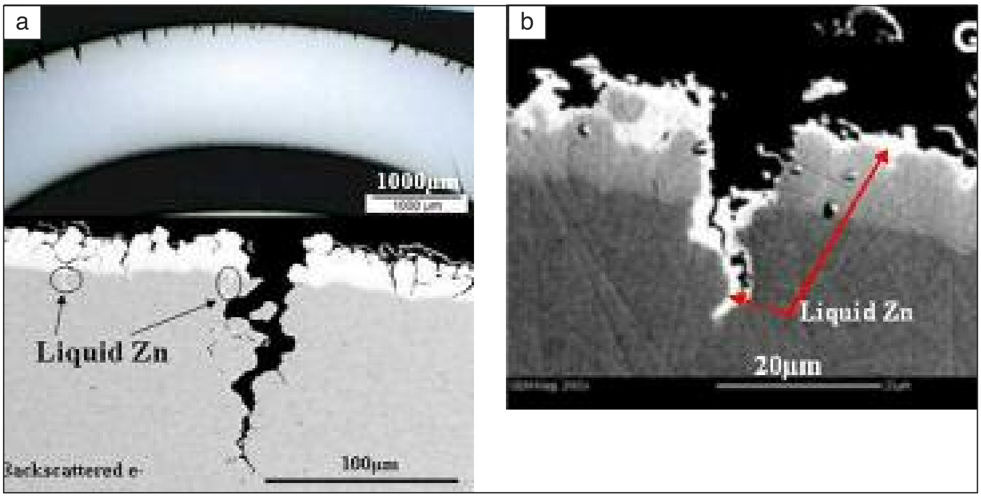


FIG. 4
(a) SEM-image of GA-coating microstructure after too short heat-treatment (remaining Fe-Zn phases), (b) SEM-image in back-scattered electrons of GI-coating microstructure after direct stamping.

(a) Immagine SEM della microstruttura del rivestimento GA dopo trattamento termico troppo breve (permangono fasi Fe-Zn), (b) immagine SEM a elettroni retrodiffusi della microstruttura del rivestimento GI dopo stampaggio diretto

the liquid Zn-bath containing a level of Al adapted to the expected coating. The coatings microstructures before and after the Hot Stamping process are illustrated by the figures 2 and 3, respectively a) for the GI and b) for the GA.
The main difference between these two coatings after the heat treatment of austenitization lies in the final coating microstructure: a high amount of Fe-Zn phase is observed on GI coatings when no more Fe-Zn phases are detected on GA coatings (Fe with Zn in solid solution).

EXPERIMENTAL PROCEDURE

All materials (22MnB5 GI and 22MnB5 GA) were sampled from coils produced by ArcelorMittal automotive coating lines. All blanks were formed to same omega shapes with a laboratory stamping process. Some real parts, made by different Hot Stampers, were also analyzed. To better understand the crack propagation mechanisms inside the coating and inside the steel, the top, the radius and the wall of the omega parts were investigated under optical and scanning electron microscopes.
The sheets were heat-treated in a furnace at 880°C (austenitization temperature) under air atmosphere, for different dwell times.

RESULTS

Macro-cracks mechanism

We observe (Fig. 4), systematically after a Direct Hot Stamping step, on GI coatings and sometimes on GA coatings (for a too short dwell time), the propagation of some big crack inside the steel (length >100 µm) in some limited areas. These areas are located on the external side of the radius (where the steel grains boundaries are under tensile stress). This macro-crack propagation is attributed to the corrosion under stress by liquid Zn or liquid Fe-Zn phase penetration inside the steel grain boundaries. In fact, some Zn phases are systematically analyzed inside the steel around the starting macro-cracks (see Fig. 4b) and the initiation site for the propagation can be also detected (see Fig. 4a).
Regarding the binary Fe-Zn phase diagram (Fig.5), it is possible to avoid these cracks at high temperatures during deformation by assuring an iron content > 70% in the coating. At a T > 782°C the phase diagram indicates that a (liquid + solid) domain exists for Fe contents lower than 60%. This value is quickly exceeded on a GA coating (no inhibition layer, start Fe% > 13%), while only a long heat treatment leads to a safe (without risk of corrosion under stress by the Zn liquid penetration) GI product.

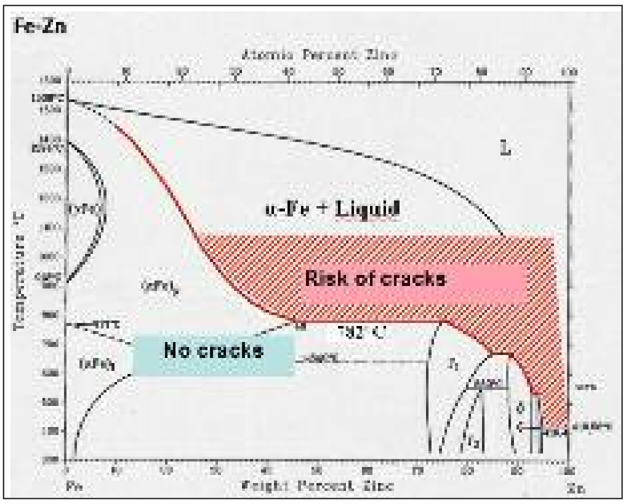


Fig. 5 ***Fe-Zn binary phase diagram shown safe and unsafe areas in terms of macro-cracks for direct hot stamping [2].***

Diagramma della fase binaria Fe-Zn che mostra le zone a rischio e quelle non a rischio in termini di macro-cricche per lo stampaggio diretto a caldo [2].

In order to obtain the deformation window for the hot stamping stage, the following laboratory trials have been conducted: Omega on a 2MN press and Hot Tensile test on a Gleeble machine. It was found that, for a temperature > 785°C, the limiting rate acceptable for hot deformation was smaller than 5%. It can be therefore considered that for the GI-coating the whole deformation has to be done at a cold stage and that the hot stage can only be used for calibration and hardening (Fig. 6). This process is an Indirect PHS Process. Moreover, on the GI coating a light shot blasting step is necessary to ensure a good phosphatizing and then a good painting adhesion.

This Indirect process leads to a production cost much higher for a component in 22MnB5 GI than for the same part in 22MnB5 AlSi, thus car makers have strong reservations about its use. Nevertheless very few automotive components showing very complex geometries cannot be directly hot stamped, for these cases, the indirect way can also be needed.

On contrary, for suitable dwell times, the GA coating can work also under a Direct Process. Moreover, it does not require any shot blasting step since a 1-3μm thick surface oxide layer allows an homogeneous phosphatizing and then a good paintability. So the second part of this paper is focused on the cracks issue encountered on this coating after a Direct PHS Process.

Micro-cracks mechanism. In order to avoid the confusion between macro and micro-cracks the following scheme was established showing the specific locations where the different types of cracks occur (Fig. 7). In fact, there are two main areas for cracks propagation inside the steel: as already mentioned, the macro-crack area is situated in the external side of the radius due to the high tensile strength while the micro-crack area is particularly situated in the wall due to a bad friction coefficient. Cracks are never observed on the top of the omega parts.

A comparison with the Al-Si coating, dedicated for main PHS applications, shows that there is no micro-crack propagation inside the steel after hot stamping operation. In fact, a soft ferrite layer of 7-10μm in thickness is developed at the steel/coating interface during the austenitization [3]. This ferrite layer has some Fe_3Al solid precipitates inside and both phases can retain the cracks propagation during the deformation due to the forming step (Fig. 8).

• **Mechanical micro-crack origin.** Due to local strong frictions (worst friction coefficient noticed on the Zn-based coatings with regards to the AlSi coating) in the walls during the Omega hot stamping the micro-cracks, always initiating in the coating, reach the steel/coating interface and propagate inside the steel in the very thin ferrite layer just under the coating (Fig. 9). These

FIG. 6
Outline for stamping 22MnB5 GI product avoiding the macro-crack propagation.
Schema per lo stampaggio di un prodotto 22MnB5 GI che eviti la propagazione di macro-cricche.

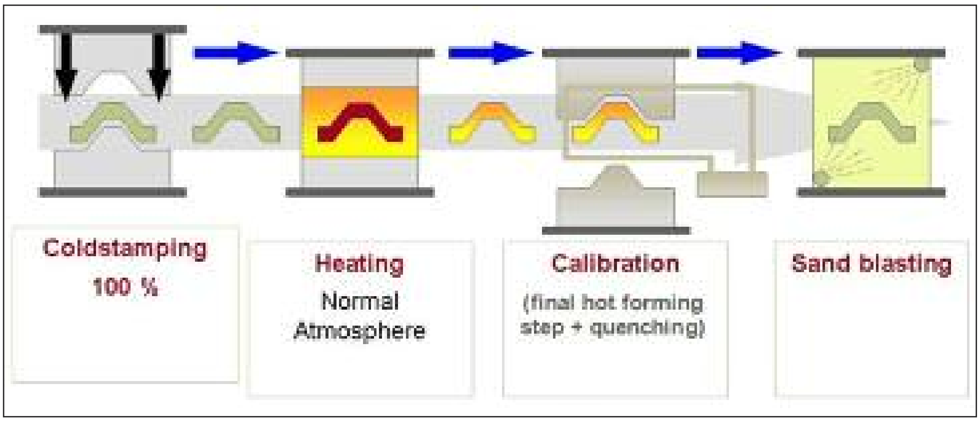


FIG. 7
Omega hot-stamped sample with different sources of cracks.
Campione di omega formato a caldo con diverse tipologie di cricche.

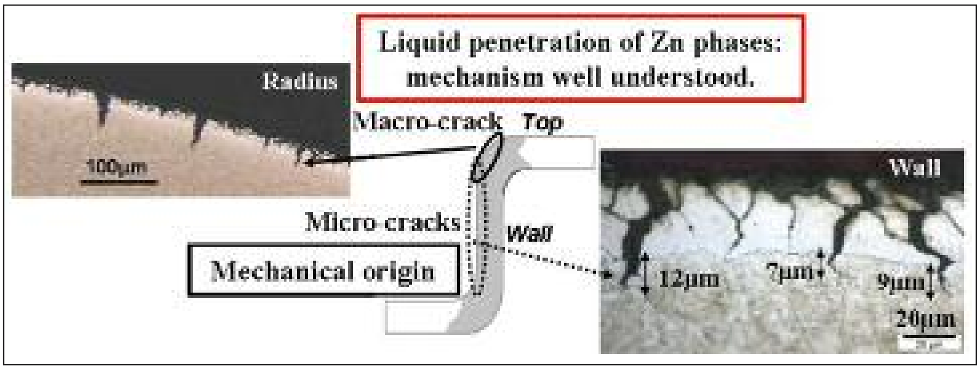


FIG. 8
Al-Si coating microstructure after press-hardening.
Microstruttura di un rivestimento Al-Si dopo incrudimento alla pressa.

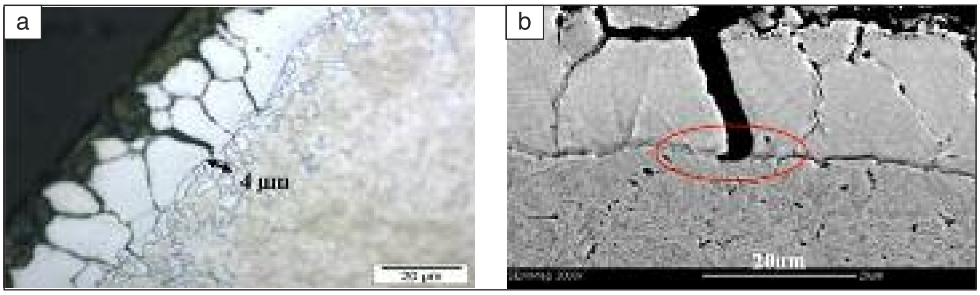


FIG. 9
(a) Micro-crack propagation inside the steel microstructure due to the presence of a thin ferrite layer under the coating, optical view, (b) Micro-crack propagation inside the steel microstructure due to the presence of thin ferrite layer under the coating, SEM-image.

(a) Propagazione di micro-cricche all'interno della microstruttura dell'acciaio dovuta alla presenza di uno strato sottile di ferrite sotto il rivestimento, vista al microscopio ottico, (b) Propagazione di micro-cricche all'interno della microstruttura dell'acciaio dovuta alla presenza di uno strato sottile di ferrite sotto il rivestimento, immagine SEM.

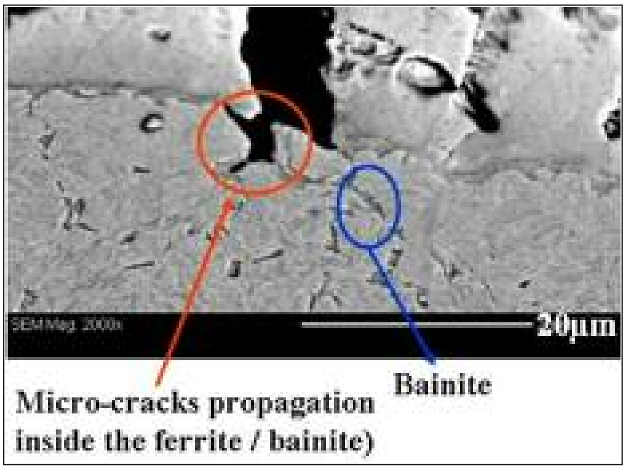


Fig. 10 ***Crack propagation inside along the martensite / ferrite interface during hot deformation.***
Propagazione di cricche all'interno lungo l'interfaccia martensite/ferrite durante la deformazione a caldo.

micro-cracks most often do not exceed 10-12µm as it is shown on the Fig. 7. But sometimes, on prototype line with a bad contact between the tools and the sheet these cracks can reach a length of 20-50 µm.

• **Sites of micro-crack propagation.** The propagation inside the steel is due to the presence of some ferritic phases in the layer just below the coating, like ferrite (and/or bainite), in the steel microstructure (Fig. 10). The cracks, initiated in the coating, continue to propagate along the martensite/ferrite interfaces. When this layer, rich in ferritic phases, is thin, like on figure 9b, the propagation is very limited (< 2 µm).
These ferritic phases, located in a layer near the steel/coating interface, have different origins. The bainite is situated in the most deformed areas. The critical cooling speed is 27°C/s for 22MnB5 without deformation. But we have established that the deformation (from 10%) induces a shift in the CCT curves to the left side and the critical cooling has to be faster ~50°C/s (Fig. 11a). The Fig. 11b shows a relationship between the deformation rate and deformation temperature. Thus, the presence of bainite depends on the cooling speed and the deformation temperature (problem of cooling speed (too low) in area showing a high level of deformation).
The Fig. 12 illustrates the steel microstructure without and with 30% of deformation, at the same temperature and cooling speed (800°C and 40°C/s).
The amount of ferrite at the interface (Widmanstaetten type), could be also increased by a small pre-existing decarburization (due to the continuous annealing step before the hot dip coating). This phenomenon is probably of small extent, we are still

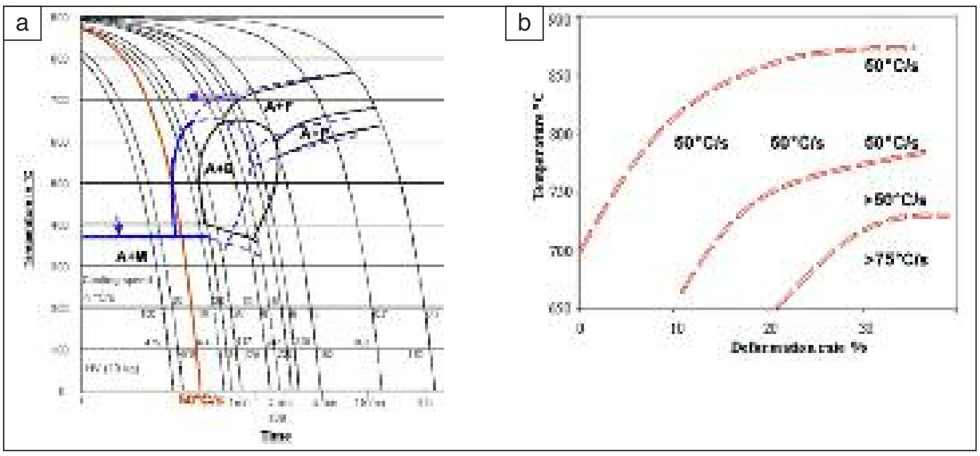
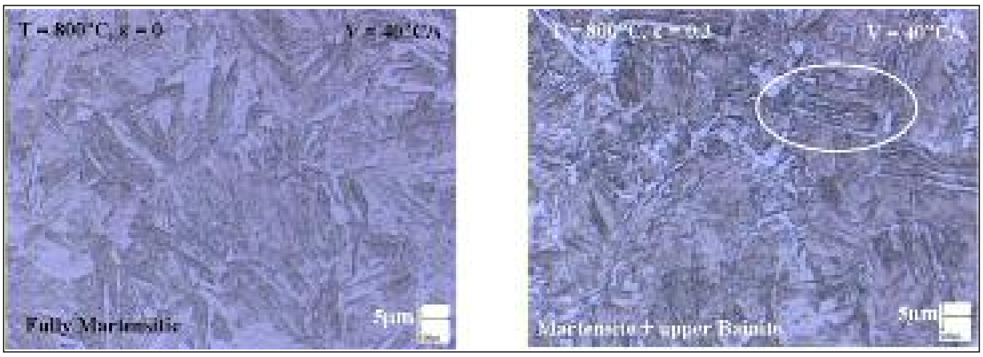


FIG. 11
(a) Shift of CCT curves under 10-30% of deformation at 800°C, Fig. 11b: Relationship between the deformation rate and the deformation temperature.
(a) Slittamento delle curve CCT per deformazioni del 10-30% a 800°C, (b) Rapporto fra la velocità di deformazione e la temperatura di deformazione.

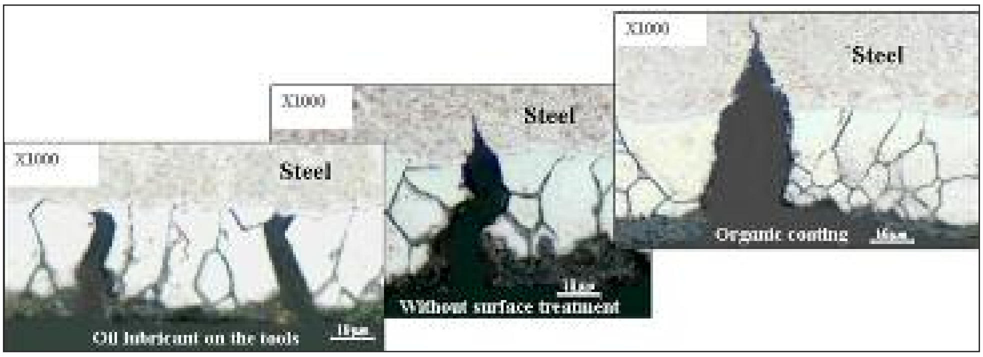
FIG. 12
Comparison of two deformation rates.
Confronto fra due velocità di deformazione.



TAB. 1
Comparison between different modes allowing avoiding the crack propagation.
Confronto fra diverse modalità che permettono di evitare la propagazione di cricche.

Substrate (1.2mm)	Coating (12µm)	Pre-treatment	HT (905°C-4mn) Crack maximum depth (µm)	
			Matrix side	Punch side
22MnB5	GA	Reference : without treatment	10	3
		Organic coating on GA surface	30	10
		Oil lubricant on the tools	0	0

FIG. 13
Comparison of GA-coating microstructures after three mode of hot forming.
Confronto fra microstrutture dei rivestimenti GA a seguito di tre modalità di formatura a caldo.



working on this point.

• **Ways to limit the micro-cracks length.** Two ways of improvement are possible. The first one is to remove the site of propagation (lattice of ferritic phases inside the previous austenitic grain boundaries). A high increase of the cooling speed after austenitization is the first solution, but it is not easy to reach this target on a Hot Stamping line. A better quenching ability could be also achieved by increasing the dwell time (and also the temperature): in fact, such a heat treatment leads to increase the austenitic grain size and then the quenching ability. But these heat treatments are not compatible with a galvanic protection. This last solution is consequently not suitable for a Zn-based coating.
The other way is to remove the mechanical origin for this propagation. It is well known that when there is a bad friction coefficient at a coating surface we transfer a high level of stress (mainly a shear stress) to the steel/coating interface. This mechanism, particularly observed in the walls, leads to cracks of 'V' shape with a length > 10 µm. A solution for this type of cracks consists in improving the tools surface friction coefficient (to apply for example a lubricant on the tools).
Some laboratory trials have been conducted to investigate this way on an omega shape. The table 1 compares three cases of using lubricant: as a reference hot-stamped 22MnB5 GA product without any special treatment, 22MnB5 GA with deposited organic coating on the GA surface and 22MnB5 GA with applied oil lubricant on the hot-stamping tools. Three corresponding GA-coating microstructures are shown on the Fig. 13. The effect of a bad friction coefficient at the surface is highlighted by the organic coating deposited at the top surface. It appears that the best solution is to apply a lubricant on the tools surface. Moreover the decreasing of the deformation at the steel/coating interface (the origin of the micro-cracks propagation) due to this better tools lubrication leads also to decrease in a great extent the level of ferrite at this location (the site of the micro-cracks propagation).
The effect of the tools lubrication has been confirmed by trials on real parts, where this step has been shown to efficiently remove the 'V' shape cracks > 10 µm and also the micro-cracks > 5 µm.

CONCLUSIONS

1) MACRO-cracks (>100 µm): these cracks are due to the corrosion under stress by liquid Zn-phases penetration in the steel grain boundaries (previous austenitic grains boundaries). So these cracks occur only when there are remaining Zn-phases inside the coating at the end of the austenitization. For this reason the GI-coating is only dedicated to Indirect hot forming process.
   On the contrary, with suitable heat treatment, the GA-coating can be dedicated to both processes: Direct and Indirect hot forming.
2) MICRO-cracks: these cracks are initiated by a mechanical issue and propagate in ferritic phases (no propagation detected inside the martenistic microstructure). On areas showing a very high friction between the steel sheet and the tools,

these cracks can reach a length of 50 µm. The best solution in order to remove these cracks is to improve the friction coefficient on the tools surface. As these cracks propagation is mainly due to the presence of some ferritic phases in a layer just situated under the coating, every improvement (like a cooling speed increasing) leading to reduce this ferrite amount will also reduce cracking sensitivity. Lubrication solution is however as such really efficient to significantly reduce ferrite formation (mainly due to the deformation).

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Abstract

Studio della propagazione di cricche nell’acciaio nel caso di acciaio temprato in pressa con rivestimento a base di zinco

Parole chiave: rivestimenti, acciaio, lavorazioni plastiche a caldo

I rivestimenti Al-Si sono la soluzione più adatta per le principali applicazioni di stampaggio a caldo, soprattutto in termini di finestra di processo per lo stampaggio a caldo e di resistenza alla corrosione perforante dopo austenitizzazione. Ma per alcuni casi specifici, alcuni clienti richiedono una protezione galvanica supplementare. Quindi, al fine di soddisfare questa esigenza sono stati sviluppati rivestimenti per stampaggio a caldo a base di zinco.

Su questi rivestimenti a base Zn è stata effettuata una completa caratterizzazione microstrutturale mirata a verificare l’influenza del rivestimento durante lo stampaggio a caldo relativamente all’interfaccia acciaio / rivestimento. Si è riscontrato che si verifica sempre propagazione di alcune cricche all'interno dell’ acciaio con rivestimenti a base Zn. Nello studio è stata fatta una distinzione fra due casi separati, corrispondenti a due meccanismi: Macro e Micro-cricche. La propagazione di Macro-cricche è legata alla penetrazione di zinco liquido all'interno dei preesistenti bordi dei grani di acciaio austenitico. Questo si verifica nelle aree che mostrano durante la deformazione un alto livello di tensioni e fasi liquide Fe-Zn residue nel rivestimento. Pertanto una deformazione a freddo è un passo preliminare indispensabile per il rivestimento GI.

La propagazione di Micro-cricche è legata a un problema di attrito ad alta temperatura tra la superficie del rivestimento e gli utensili. La maggiore densità di micro-cricche è quindi osservabile sulle aree più sensibili all’ attrito. Nell’ indagine sono state identificate all'interno dell’acciaio le fasi responsabili di questa propagazione.

Sono state proposte alcune soluzioni per evitare questi fenomeni, in particolare nel caso Micro-cricche per applicazioni di stampaggio a caldo diretto (rivestimenti GA).