Fatigue Resistance of Hot-dip Galvanized Hot-rolled and High-Silicon TRIP Steel

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Abstract: Automobile industry has to cope with the emergence of high strength steels, as TRIP steel. Hot-dip galvanization is the most cost-effective technique to protect steel against corrosion. It consists in dipping steel parts in a molten zinc bath at 450 centigrade. A reduction of fatigue endurance has been noticed. Our study aims to point out the mechanisms responsible for this degradation. Experimental tests were carried out so as to investigate two assumptions of embrittlement: the cracking from the zinc coating layers and the modification of the mechanical properties during the heat treatment in melted zinc. The main criterion was the fatigue lifetime. Our results show that:

- The cracks in the coating are mostly stopped at the zinc-steel interface.
- The heat treatment increases the fatigue resistance.

The heat treatment is not responsible for the lifetime drop. The cracks in the zinc coating may not cause the fracture.

Key words: hot-dip galvanization; TRIP steel; fatigue properties

1. Introduction

Automobile industry current ecological and economical preoccupations make essential to develop new materials, able to provide lightweight improvement. Advanced High Strength Steels aim to meet this requirement, with both interesting mechanical resistance and deformability properties. TRansformation-Induced Plasticity (TRIP) steel, with an ultimate stress tensile between 600 and 1000 MPa and a total elongation up to 30%, is one of them. TRIP steel presents a multiphased microstructure at room temperature, as a result of a complex thermomechanical route: ferrite, retained austenite and eventually martensite and bainite. However, like many other steels, TRIP steel is vulnerable to corrosion. Zinc coating protection is used to make up for it. Several processes exist and the most common are hot-dip galvanization, continuous galvanizing, matoplasty, electrolytic galvanizing and sherardizing. Galvanizing manufacturer guarantees galvanization life as a function of coating thickness, which varies according to the process. Thus, hot-dip galvanization is the most cost-effective technique to ensure automobile steel parts protection. Hot-dip galvanization takes place in two steps.

Firstly, steel part surface is prepared with pretreatment baths: degreasing, rinsing, pickling, rinsing, fluxing and drying. Secondly, it is dipped in a molten zinc bath for galvanization. Hot-dip galvanization main parameters are bath composition, temperature and treatment time. Since automobile parts undergo cyclic loading, it is important to check, if the fatigue performance of the steel will be modified by hot-dip galvanization.

Hot-dip galvanization can possibly result in a reduction of fatigue strength for most steels [1].In this paper, hot-dip galvanization effect, on hot-rolled TRIP800 steel fatigue properties, was discussed. The purpose was to determine, whether hot-rolled TRIP800 fatigue behavior changes after hot-dip galvanization and to point out, if need be, the potential linked mechanisms. In this aim, heat-treatment, due to the stay in hot liquid zinc bath, and zinc coating influences were studied.

2. Method

2.1 Material

Here, hot-rolled TRIP800 steel was studied. This steel grade contains high silicon content. The role of the silicon is to stabilize retained austenite at room temperature and to inhibit cementite precipitation during bainitic holding. Hot-rolled, high-silicon, TRIP800 contains 0.25 % wt. carbon, 2 % wt. silicon and more than 10% retained austenite. Sheet thickness is 3 mm. Hot-dip galvanization is made at 440°C, during 4 min in a Galvacar® zinc bath. The average thicknesses of zinc coating for the hot-dip galvanized samples were about 80µm.

The geometry and dimension of samples were chosen, so that the fracture will be in the gauge length (Fig. 1). The tensile direction was chosen perpendicular to the rolling direction for all the specimens.

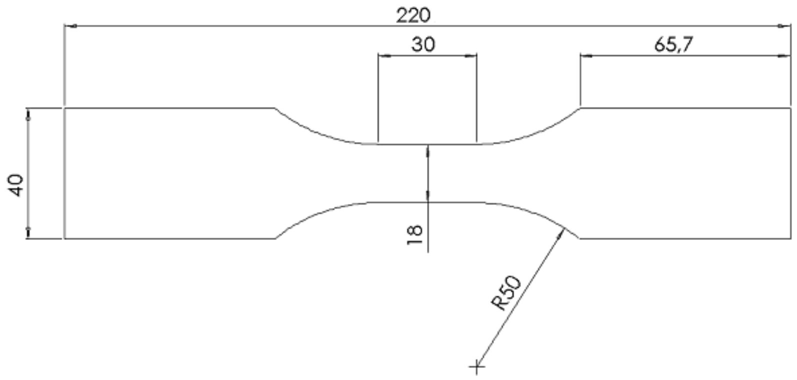

Figure 1: Sample configuration for mechanical testing

Hot-dip galvanization of the steel in hot liquid zinc induces a heat-treatment. So as to analyze its influence, we chose to reproduce the same heat-treatment conditions as for the hot-dip galvanized TRIP800 steel: bath temperature of 440°C, immersion time of 4min, immersion velocity of 1.5m/min and emersion velocity of 0.5m/min. Heat treatment was performed in alkaline molten salt bath, which provide a homogeneous temperature. Immersion temperature was checked by type K (chromel–alumel) thermocouple. Heat-treatment experimental device is shown in figure 2.

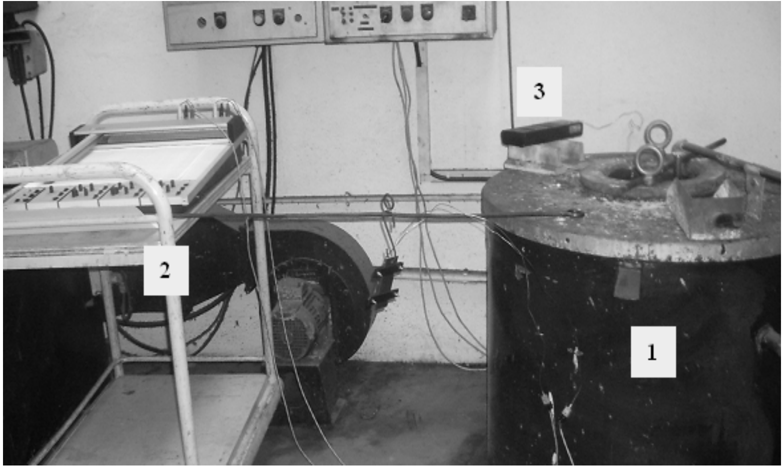

Figure 2: Experimental device for the heat treatment operation

(1: molten salt bath furnace, 2: plotter, 3: digital thermocouple thermometer)

In this way, three kinds of TRIP800 samples, with identical geometry, were obtained: bare steel with no treatment, hot-dip galvanized steel and heat-treated steel.

2.2 Mechanical testing

Mechanical tests were performed, in order to determine steel behavior modification after hot-dip galvanization. Quasi-static tensile tests aimed to define mechanical properties of the different samples. These uniaxial tensile tests were performed at room temperature. The testing equipment was an Instron electromechanical test machine with maximal capacity of 250 kN. Tensile tests were quasi-static, since the applied strain rate was 10^{-3} s^{-1}. The tensile direction was perpendicular to the laminar direction.

Fatigue behavior was an important criterion is this study. Uniaxial tensile fatigue tests were performed on both bare and hot-dip galvanized samples. The testing equipment is a servohydraulic Schenck device able to apply axial loads up to 100 kN. All tests were carried out at room temperature. The tensile direction was the same as for static tests. A sinusoidal load wave was applied with a frequency of 30 Hz and a strength rate of 0.1. After cyclic loading, SEM analyses were performed on fracture facies and polished samples.

Standard TRIP800 steel samples were made through cutting and polishing for hardness tests. The Vickers machine test indenter was a 136 degrees square-based diamond pyramid. Average core hardness measurements were done under a load speed of 70µm/s, a loadtime of 15s and a load test of about 0.196 kN (20kgf).

3. Results

3.1 Mechanical properties

Bare, hot-dip galvanized and heat-treated hot-rolled TRIP800 steels were tensiled, in the above-mentioned quasi-static conditions. The mechanical properties are presented graphically in figure 3.

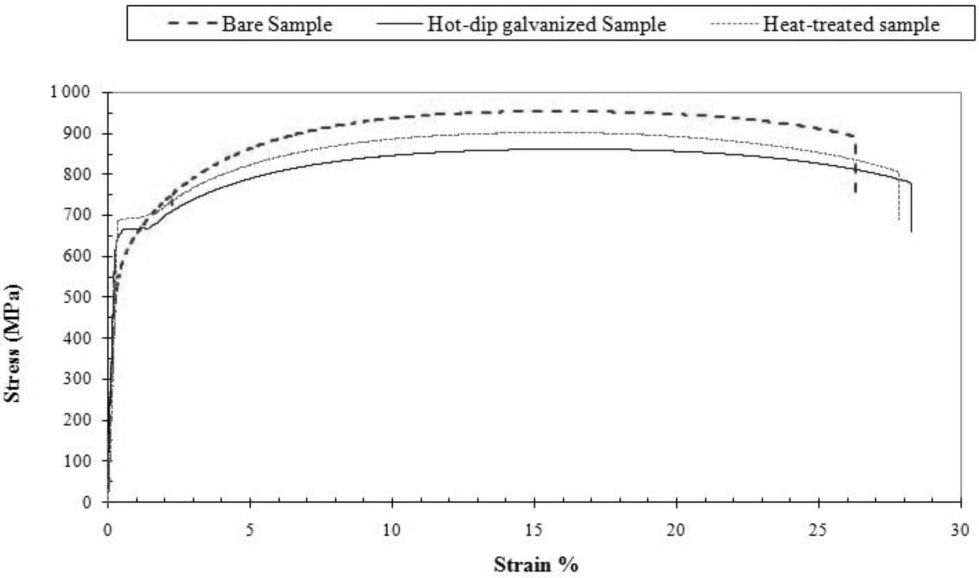

Figure 3: Stress-strain curves of hot-rolled high-silica TRIP800 steel

Hot-rolled TRIP800 bare steel has elastoplastic behavior in uniaxial tension. Hot-dip galvanized and heat-treated steels both present a Lüders plateau. The Lüders plateau is caused by a localized plastic deformation, called Lüders band: it results from inhomogeneous yielding. Ultimate tensile stress, yield stress and total elongation at fracture are different for bare (954MPa, 573MPa and 25.8%), hot-dip galvanized (862MPa, 664MPa and 27.9%) and heat-treated steels (901MPa, 690MPa and 27.4%). So, hot-dip galvanization modifies material behavior of bare steel: 15% increase in yield stress, 9% drop in ultimate tensile stress. Moreover, it seems that hot-dip galvanization increases total elongation at fracture for several tested specimens.

The used experimental device (Fig. 2) was validated according to the good matching between the static behaviors of heat treated steel and hot-dip galvanized steel. Indeed, hot-dip galvanized and heat-treated specimens present a reduction in ultimate tensile stress and an enhancement of ductility.

Furthermore, average core hardness measurements don’t show an important variation for the studied cases: the average core hardnesses of bare, hot-dip galvanized and heat-treated steels were respectively 262, 253 and 254 HV20. In spite of a low evolution of core hardness after galvanization, it can be concluded, that these results correlate with tensile tests, since ultimate tensile stress value is more important for bare steel, than for hot-dip galvanized and heat-treated steels.

3.2 Fatigue properties

After cycling loading, Wöhler curves of bare, hot-dip galvanized and heat-treated TRIP800 steel were built. These curves display the applied stress range versus the logarithmic number of cycles to failure (Fig. 4).

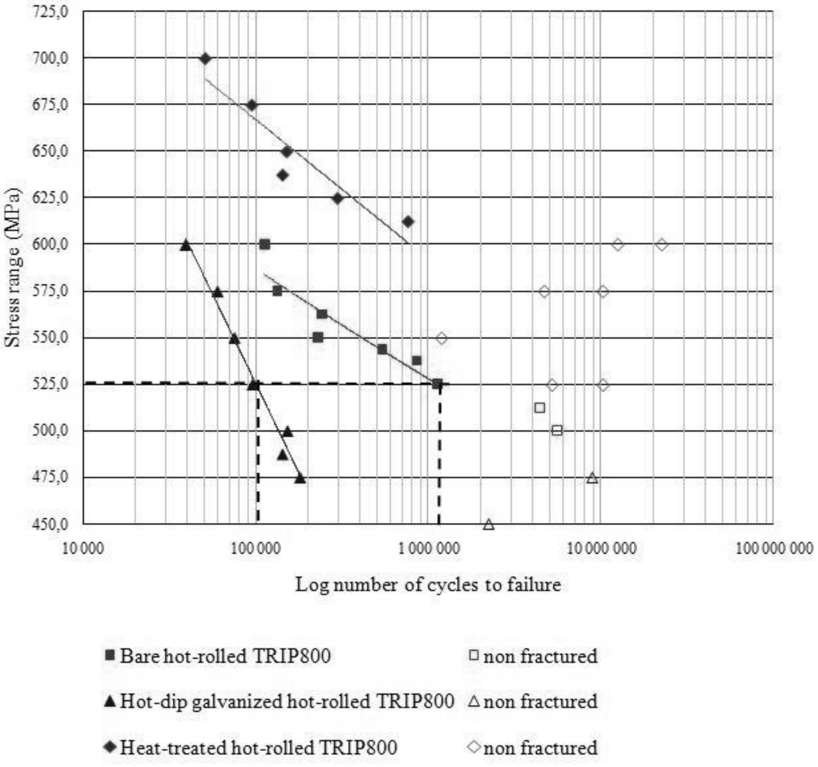

Figure 4: Wöhler curves of hot-rolled high-silica TRIP800 steel for hot-dip galvanized, bare and heat-treated steel

For a same stress range of 525 MPa, the lifetime of the hot-dip galvanized TRIP800 steel is about 100 000 cycles, whereas bare steel lifetime is about 1 000 000 cycles, and heat-treated steel do not break.

3.2 Cracking from zinc coating

As a result of hot-dip galvanization in liquid zinc, a multiphase zinc coating is metallurgically bound to the steel substrate. During immersion in the zinc bath, a double diffusion reaction occurs between iron and zinc. Thus, the coating is alloyed and multilayered. Thicknesses of this intermetallic layers depend especially on immersion time and temperature. Outer layer, called η, is pure zinc. Between η and steel substrate, there are three intermetallic phases with different morphology and composition [2]:

- The zeta layer, ζ, is the richest in zinc after η: the zinc content is approximately 92.9 - 94.1 at. %. This thick layer grain structure is not well defined.
- The delta phase, δ, has a zinc composition of 86.8 - 91.9 at. %. δ layer presents columnar grains, which growth is perpendicular to the steel substrate surface. It can be divided into two morphologies: δ_P, palisade morphology on the zinc rich side, and δ_K, compact morphology on the iron rich side.
- The gamma phase, Γ, with an iron content of 69 - 82 at. % is the very thinnest layer and is close to the zinc-steel interface. This phase is not always visible.

Cracks are found in the more brittle layers ζ and δ. They are caused by thermal contraction, in steel and coating, occurring during cooling after hot-dip galvanization. The difference between the linear expansion coefficients of the intermetallic layers and the steel, explain these thermal coating stresses (12.10^{-6} K^{-1} for zinc and 26.10^{-6} K^{-1} for steel) [4].

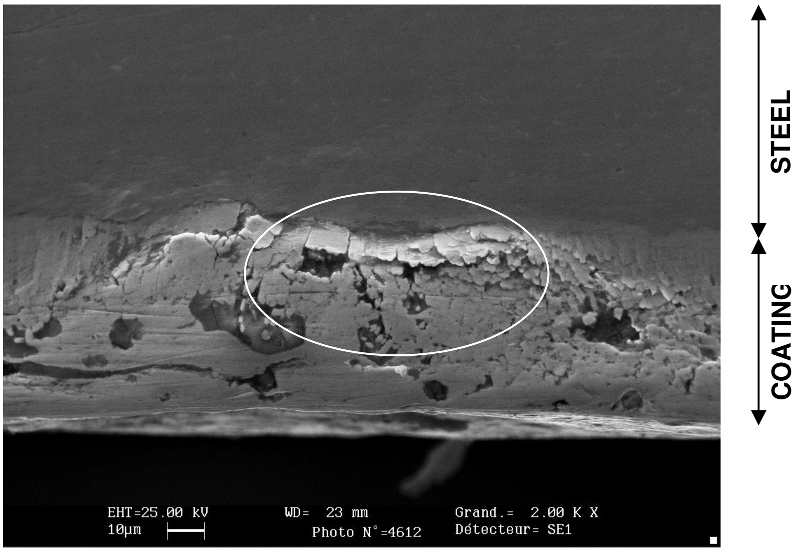

Figure 5: Cracks in the coating are stopped at zinc-steel interface after fatigue test

The investigation of crack propagation, from initial cracks in zinc coating to steel substrate, was carried out on TRIP800 steel, after cycling loading. The performed SEM analyses show that the cracks are confined in the coating after fatigue test (Fig. 5).

4. Discussion

4.1. Heat treatment, induced by immersion in zinc liquid

While being dipped in the molten zinc bath, the steel parts are heat-treated. According to Li and Wu’s [3] Continuous Cooling Transformation curve, for a hot-rolled TRIP steel at 440°C, during 4 min, a transformation from austenite to bainite can occur. Although, the high content in silicon, in hot-rolled TRIP800 steel, should limit this phenomenon, we can assume that such a transformation is possible and as other transformation. Here, the hot-rolled TRIP800 steel presents a ferritic matrix, bainite, martensite and retained austenite.

The obtained stress-strain curves (Fig. 3) show a Lüders plateau for heat-treated and hot-dip galvanized TRIP800 steel and the core hardnesses are identical. Therefore, the microstructure modification is confirmed. The microstructure modification in core hot-dip galvanized steel is analogous to heat-treated steel: yield strength and ductility are improved, in spite of a light drop in ultimate tensile strength.

Nevertheless, fatigue properties of the two kinds of specimens are really different. Indeed, their effects are the opposite: hot-dip galvanizing reduces lifetime, while heat-treatment increases fatigue resistance. For a same stress range of 525 MPa, the lifetime of the hot-dip galvanized TRIP800 steel drops by a factor of ten in comparison with bare steel, which is quite important. In fact, hot-dip galvanized Wöhler curve should be compared to heat-treated curve, because both specimens are heat treated. In this manner, the harmful effect of hot-dip galvanization is obvious.

4.2. Crack propagation from coating to steel substrate

On one hand, Bergengren and Melander [5] present results for a high strength steel (3mm thick, 680 MPa yield strength): fatigue degradation is explained by cracking from coating cracks and coating thickness influence is studied. Moreover, for their steel, the thicker coating gave the shorter life time (coating thickness of 80,125 and 225µm). De la Cruz and Ericsson [4] state a fatigue lifetime decrease in 9% after hot-dip galvanization (hot rolled bar 20mm in diameter, yield strength 779MPa). They account it by either a cracking from δ and ζ layers or hydrogen embrittlement after pickling.

On the other hand, Nilsson et al.[6] found that the coating thickness (80-185µm) has no obvious influence on the fatigue properties on another hot-rolled high strength steel (5-6mm thick, yield strength 690MPa,). The reduction in fatigue limit was 35%. According to Nieth and Wiegand’s [7] paper, hot-dip galvanization has no adverse effect on fatigue properties , for a high strength steel (8mm, yield strength superior to 600MPa) with a coating between 80 and 100µm.

Vogt et al.[8] noticed that the thermal pre-existing crack pattern is not involved in fracture damage of hot-dip galvanized steel and a crack can propagate in the steel substrate provided that the total coating is cracked (sheet 1.42mm thick, yield strength 330MPa, coating thickness 58-102µm).

For the studied hot-dip galvanized TRIP800 steel, the cracks from δ and ζ layers were confined in the coating and probably stopped at zinc-steel interface.

Reumont et al. [9] made tensile tests in air on hot-dip galvanized interstial free steels. From previous studies, they conclude that pre-existing thermal cracks propagate through the coating under increasing loading and stoopped at zinc-steel interface. The propagation grows initially in mode I and develops in mode II at the interface. The pre-existing cracks in the coating may not be responsible for the fatigue performance degradation.

4.2. Possible embrittlement during surface preparation prior to hot-dip galvanization

Scanning electron microscopy provides a strong indication of the probable cause of failure: failure analysis tries to connect failure appearance and failure cause. A multiphase steel presents generally mixed fracture modes. Indeed austenite rupture is ductile, due to the numerous slip systems of the fcc structure. On the contrary, ferrite, martensite and bainite exhibit sensitivity to brittle rupture by cleavage [10]. Fracture facies analyses were carried out on hot-dip galvanized and bare steel samples after fatigue test. In the fatigue area, identified by fatigue striation (Fig. 5), the rupture seems transcrystalline close to zinc-steel interface (Fig.6), contrary to uncoated steel, where the rupture was rather intercrystalline. The cracking probably initiated at this interface. Reumont et al. [9] noticed a transgranular brittle rupture, for a hot-dip galvanized interstitial free in NaCl environment. It was associated with hydrogen embrittlement. Moreover, it is well known that high strength steels are particularly sensitive to hydrogen embrittlement.

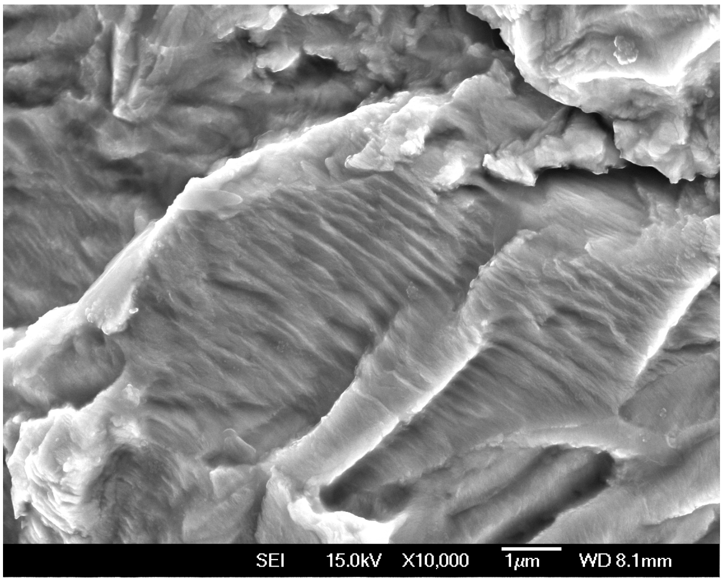

Figure 5: fatigue striation in hot-rolled TRIP800 steel after cycling loading

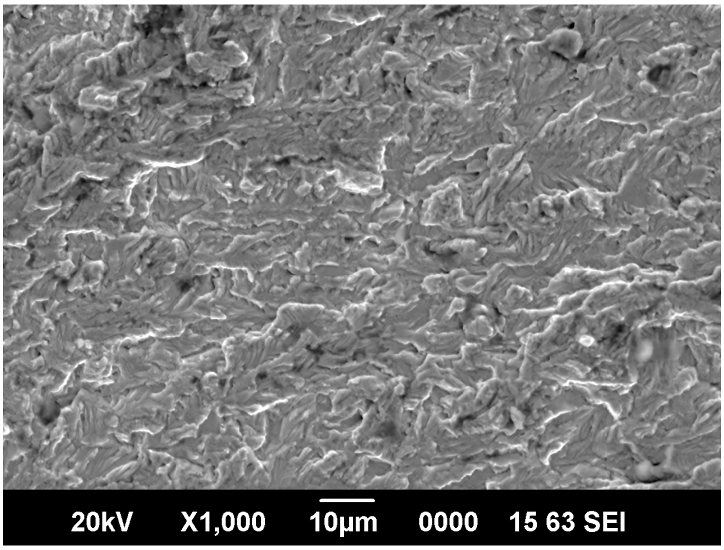

Figure 6: Micrography of a fracture facies (fatigue area) close to the zinc-steel interface

Carpio et al. [11] study the influence of the surface preparation operations on mechanical properties. Hydrogen content measurements, after each operation, make them assume that some hydrogen could concentrate at coating intermetallic layers surfaces. It can be a possible mechanism for TRIP800 steel embrittlement, after hot-dip galvanization. Further investigations are under study, so as to elucidate this assumption.

5. Conclusion

(1) Hot-rolled TRIP800 steel is quite sensitive to hot-dip galvanization. Indeed, for a stress range of 525Mpa at a load ratio of 0.1, the number of cycles to failure is drop by a factor of ten compared to uncoated steel. Both quasi-static and fatigues properties are affected: drops in ultimate tensile stress and fatigue resistance.

(2) Assumption of hot-rolled TRIP800 steel embrittlement by heat-treatment, occurring during 4min immersion, of hot-rolled TRIP800 steel part, in molten zinc at 440°C, was invalidated. On the contrary, heat-treatment increases hot-rolled TRIP800 properties, mainly due to ductility enhancement. Average hardness measurements and quasi-static tensile tests show similar behavior for hot-dip galvanized and heat-treated steel (Lüders plateau, same core average hardnesses). So, it can be concluded, that core microstructure modification is not involved in hot-rolled TRIP800 embrittlement after hot-dip galvanization.

(3) Cracking investigation in zinc coating reveals that the cracks from delta phase are stopped at zinc-steel interface, after cycling loading. As the final crack pattern

does not match the thermal pre-existing one, some papers put the question of a possible hydrogen embritllement after surface preparation.

6. Acknowledgements

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7. References

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