EFFECT OF Ti AND Nb ADDITIONS ON THE FORMATION OF CRATERS FOR IF STEEL GALVANNEAL COATINGS[1]

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Abstract

Interstitial Free steels feature alloying elements present in an ultra low C matrix that account for C stabilization and consequently a high number of C free interstitials in a ferrite matrix that give these steels the ideal texture, low yield point, favourable plastic strain ratios, high elongation and n-value necessary for an ideal performance on forming press, drawing and stamping operations especially for automotive body part applications. The purpose of this technical paper is to propose that Ti-stabilized IF steel substrates are more reactive than dual-stabilized IF steel substrates due to the presence of islands of Nb oxides at the interface between the steel substrate and the GA coating. These Nb oxides block the Fe-Zn interdiffusion along the ferrite grain boundaries. Hence, higher reactivity rates for Ti-stabilized IF steel grades mean that this substrate is more prone than a dual-stabilized IF steel to the formation of outbursts and consequently the formation of craters, which is morphologically characterized as a cluster of outbursts whose formation mechanisms are based on capillarity effects accounting for amounts of liquid Zn being drained away from these spots. Besides, this work has also explored the typical chemistry and morphology features of craters on top of dark and light streaked areas on GA coatings, showing that there are δ crystals on the bottom of craters on light streaked areas, whereas Γ phase was found on the bottom of craters on dark streaked areas. Therefore, it turns out that craters on dark streaked regions are deeper than those on light streaked areas. On top of that, the crater coverage on dark streaked regions is larger than on light streaked areas.

Key words: IF steels; Galvannealing; Craters

EFEITO DAS ADIÇÕES DE Ti E Nb NA FORMAÇÃO DE CRATERAS NA SUPERFÍCIE DE AÇOS IF GALVANNEALED

Resumo

Os aços Interstitial Free contêm elementos de liga em uma matriz de aço ultra baixo C responsáveis pela estabilização do C e, conseqüentemente, um número elevado de interstícios em uma matriz ferrítica conferindo a estes aços uma textura ideal, baixo limite de escoamento, deformação plástica favorável, alto alongamento e coeficiente de encruamento necessário para uma boa performance nas prensas hidráulicas e operações de estampagem principalmente para aplicações automobilísticas. O propósito deste trabalho é propor que os substratos de aços IF estabilizados ao Ti são mais reativos que os substratos de aços IF estabilizados ao Ti e Nb devido à presença de ilhas de óxidos de Nb na interface entre o substrato do aço e o revestido GA. Estes óxidos de Nb bloqueiam o interdifusão de átomos de Fe e Zn ao longo dos contornos de grãos de ferrita. Por esta razão, altas taxas de reatividade nos aços IF estabilizados ao Ti significam que este substrato de aço é mais propenso à formação de outbursts do que os substratos de aços IF estabilizados ao Ti e Nb, e conseqüentemente à formação de crateras. As crateras, por sua vez, são morfologicamente caracterizadas como regiões de aglomeração de outbursts cujo mecanismo de formação baseia-se nos efeitos de capilaridade responsáveis pela drenagem de Zn líquido para fora destas regiões. Adicionalmente, este trabalho também explora a composição química típica e a morfologia das crateras em regiões de faixas (streaks) claras e escuras, mostrando que existem cristais da fase intermetálica δ no fundo de crateras em regiões claras, enquanto cristais da fase intermetálica Γ foram encontrados no fundo das crateras nas regiões escuras. Portanto, as crateras em regiões escuras são mais profundas do que crateras em regiões claras. Finalmente, as áreas cobertas por crateras em regiões escuras são superiores às das regiões claras.

Palavras-chave: Aços IF; Galvannealing; Crateras.

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1 INTRODUCTION

The industry uses Ti-, Nb- and dual-stabilized IF grades for a variety of applications. These grades are easy to galvanise as the bulk of the substrate is ferrite and preparation of the substrate surfaces is such that their wettability by liquid Zn-Al alloys becomes trivial. However, it has been noticed that Ti-containing grades do not behave as well as Nb-bearing grades when manufacturing galvannealed coatings. The former usually exhibit features of decreased coating integrity (i.e. decreased coating adherence) and a surface defect whose appearance is crater-like, which can be characterized as a discontinuity on the surface of galvanneal coatings and results in poor coating appearance. These problems are not commonly seen on the surface of Nb-stabilized IF steel coatings. However, Nb-bearing grades do not generally have the same desirable mechanical properties (particularly with respect to formability) found in Ti containing grades. The former are generally prone to have higher alloying costs requiring higher power to recrystallize anneal and galvanneal. Therefore, dual-stabilized IF steel grades were developed as a compromise between these two behaviours. The differences between these two grades in the galvannealing process are remarkable considering the rather low level of alloying elements in these steels and the fact that the Ti and Nb additions are 'tied up' as nitrides, sulfides, carbo-sulfides and phosphides to a large extent. However, it is believed that the fundamental origin of the differences between the process behaviour of these steel substrates lies in a reasonable comprehension of the effect of Ti and Nb additions upon steel substrate reactivity.

2 LITERATURE BACKGROUND

Several authors have assessed the main effects of alloying elements upon the GA coating properties, such as Marder, Jordan and O'Dell, among others. Nb has been reported improving powdering resistance and resistance to cold-work embrittlement, reducing planar anisotropy and leading to the development of a preferred texture for good deep-drawability. However, one of the drawbacks of Nb additions is that it raises the recrystallization temperature and consequently a higher annealing temperature is required to achieve full recrystallization. Besides, it has been reported that a Nb-added IF steel has difficulties to obtain the excellent and homogeneous mechanical properties through the front to the tail of a coil.

Ti, on the other hand, has been known for its improved overall mechanical properties, especially formability, but it shows poor powdering resistance. The formation of Ti precipitates leads to the stabilization of elements known to segregate to ferrite grain boundaries - therefore blocking Fe-Zn interdiffusion - such as C, N, S and P. Due to the high Ti affinity for N, Ti nitrides are the first precipitates to form regardless of the IF steel substrate chemistry and atomic ratio. In the end, after a sequence of S, C and P precipitation, the ferrite grain boundaries are clean for the diffusion of Zn atoms down from the Zn coating and Fe atoms up from the steel substrate. The degree of cleanliness - or reactivity - of an IF steel substrate is expressed by the amount of excess Ti in solid solution, i.e. the final Ti content that is not tied up with C, N, S or P, and therefore is available in solid solution for further diffusion. A positive excess Ti indicates that the ferrite grain boundaries are clean for Fe-Zn interdiffusion, whereas a negative value

means that not all C, N, S and P have been stabilized, therefore segregating to ferrite grain boundaries and blocking Fe-Zn interdiffusion.
All in all, most part of authors have shown that Ti-only-stabilized IF steels are more reactive and prone to outburst than dual-stabilized IF steel substrates, but to date there is no conclusive reason to explain why this takes place in the bulk of the ferrite steel substrates.

3 EXPERIMENTAL PROCEDURE

Two IF steel grades were used for this technical paper: a Ti-only-stabilized and a dual-stabilized grade. The chemistry for these IF steel grades are shown according to Figure 1. The samples were sheared to fit the standard size for the Hot-Dip Process (HDP) Simulator, i.e. 120 x 200 mm. A Jobin-Yvon/Horiba Quantum IQ V2.22 Glow-Discharge Optical Emission Spectroscope was used as a tool to obtain the surface chemistry profiles of the alloying elements for the as-received samples.

wt% C	0,003
wt% Mn	0,16
wt% P	0,008
wt% S	0,008
wt% Si	0,007
wt% Cu	0,02
wt% Ni	0,013
wt% Cr	0,02
wt% Mo	0,004
wt% V	0,002
wt% Nb	0,006
wt% Ti	0,052
wt% Sn	0,002
wt% Al (sol.)	0,03
wt% N	0,002

wt% C	0,002
wt% Mn	0,19
wt% P	0,005
wt% S	0,005
wt% Si	0,005
wt% Cu	0,018
wt% Ni	0,019
wt% Cr	0,02
wt% Mo	0,001
wt% V	0,001
wt% Nb	0,029
wt% Ti	0,02
wt% Sn	0,006
wt% Al (sol.)	0,035
wt% N	0,003

Figure 1. Chemistries for the Ti-stabilized IF steel grade (left-hand side) and the dual-stabilized IF steel grade (right-hand side).

In order to simulate the operating conditions of a commercial Continuous Galvanising Line (CGL) in a smaller scale, an Iwatani-Europe Hot-Dip Process Simulator was used. This equipment is composed of an upper chamber for sample input/output and further cooling by fast rates. The infrared heat treatment furnace lies in the middle of the equipment, operating by either radiant or induction heating. Finally, the lower middle part of the equipment consists of a chamber for slower cooling rates, which is placed right above the 30 kg Zn bath crucible.
In order to set up the heat treatment cycles for both IF steel grades, with a special focus on the galvanneal (GA) heat treatment parameters - GA temperature and soaking time -, tentative dipping samples were run at the HDP simulator aiming to produce a fully-developed GA coating on top of the steel substrate by using the same effective Al 0.13 wt% and same furnace controlling atmosphere 95% N_2 + 5% H_2. The setting or target condition was to produce a 1 μm-thick Γ layer close to the interface between the Zn coating and the steel substrate. Thermal profiles for both Ti- and dual-stabilized IF steel substrates are shown according to Figure 2.

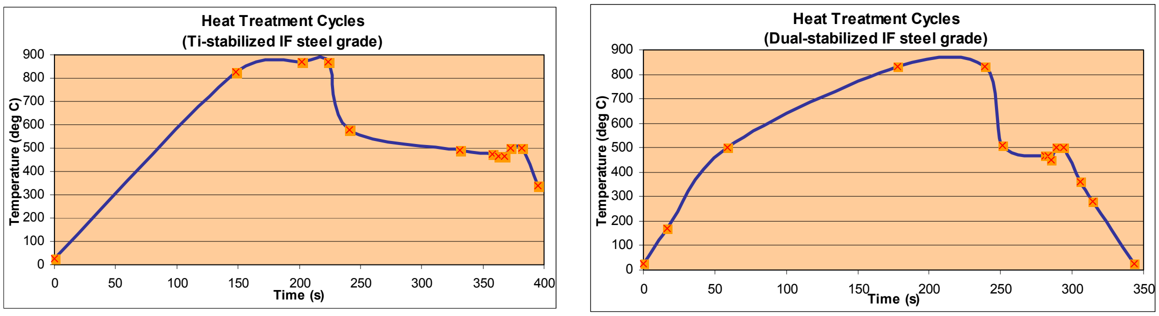


Figure 2. Thermal profiles set up for both IF steel grades at the HDP simulator.

Once the thermal profiles for both IF steel grades were properly set up, interrupted cycles were run for bare samples from both IF steel grades, stopping the cycle at pre-determined spots along the thermal curve, and samples were taken at each one of these spots along the thermal profile. The purpose of this procedure was to observe how Ti and Nb surface concentrations evolved along the thermal profile for Ti- and dual-stabilized IF steel grades compared with the as-received condition as well as to assess the effect of the annealing and galvannealing heat treatments upon the Ti and Nb surface segregation.

A Philips SEM 515 Scanning Electron Microscope was used for image analysis, attached to a Link Analytical Pentafet Energy Dispersive Spectroscope microanalyser, used as an aid for a qualitative approach to identify the Fe-Zn intermetallic compounds through the GA coating. SEM was also used as a tool for image analysis on samples with streaks on top of GA coatings as well as its attached EDS microanalyser for the chemistry measurements. A new set of GD-OES profiles were run for the coated samples, this time aiming to identify the Fe-Zn intermetallic phases, to determine the thickness of the Γ layer and also to obtain the Ti and Nb concentration profiles through the GA coating, at the interfacial layer and into the steel substrate.

After that, an X-ray Photoelectron Spectroscopy PHI Quantera SXM was used to identify the chemical state of Nb at the surface of IF steel substrates.

The samples with streaks on top of GA coatings were supplied by two different suppliers. In order to establish comparisons between the areas featuring dark and light streaks, SEM was used for image analysis and EDS for chemistry measurements.

Finally, new interrupted thermal cycles were set up for the HDP simulator, this time featuring special cooling conditions, by means of holding much faster cooling rates inside the He cooling chamber right after the GA heat treatment. This procedure was performed in order to replicate the ideal operating conditions in a CGL for crater formation. Both Ti- and dual-stabilized IF steel panel samples were ground, polished and then set up in the HDP simulator to undergo annealing under regular time and temperature conditions for both IF steel substrates, dipped in the Zn bath and underwent GA heat treatment under the new operating conditions, stopping the GA heat treatment at 0s, 2s and 4s, and then the panels were submitted to faster cooling rates than the previous procedures. Figure 3 below features the thermal cycles for both steel substrates.

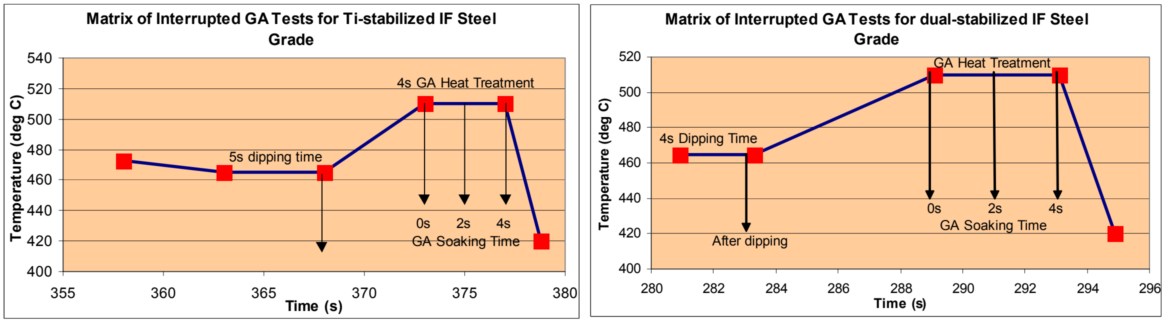


Figure 3. Matrix of interrupted GA thermal cycles for both IF steel grades at the HDP simulator.

4 RESULTS AND DISCUSSION

The trial dipping samples ran at the HDP simulator in a 0.13 wt% effective Al in Zn bath and same furnace controlling atmosphere 95% N_2 + 5% H_2 showed that, for a Ti-only-stabilized IF steel substrate, 8s at 510 ^{0}C were necessary to produce a fully-developed GA coating featuring a 1 μm-thick Γ layer. On the other hand, 15s were needed for the dual-stabilized IF steel substrate to produce the same results at the same temperature, according to images shown on Figure 4. Therefore, it can be concluded that a Ti-stabilized IF steel substrate is more reactive than a dual-stabilized IF steel substrate.

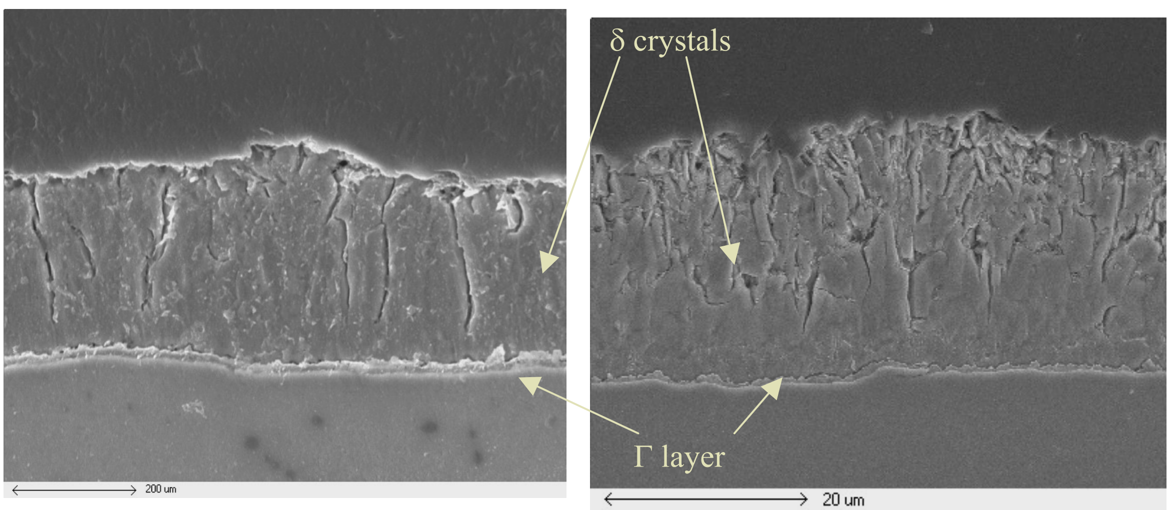


Figure 4. SEM images at 2100x and 2020x featuring the GA coating cross section for the Ti-stabilized IF steel grade (left-hand side) and the dual-stabilized IF steel grade (right-hand side).

The GD-OES results for bare samples on both IF steel grades showed that there is a Ti depletion at the outermost surface of both Ti- and dual-stabilized IF steel substrates. The influence of annealing and galvannealing heat treatments upon Ti surface segregation is minimal and not remarkable. After a sputtering depth of 0.02 μm into the steel substrate, a Ti depletion was found on most samples along the thermal profile for both IF steel grades. Moving farther into the steel substrate - 0.1 μm – a slight Ti enrichment was found for most part of samples on both IF steel substrates. This enrichment is likely to be associated with a small amount of Ti that has not been previously tied up, therefore available in solid solution. According to excess Ti calculations based on the formula excess Ti = total Ti – 3.99C – 1.49S – 3.42N – 1.55P given by Marder et al., there are 90 ppm Ti left in solid solution and available for diffusion prior to the annealing heat treatment on the Ti-only-stabilized IF steel grade. GD-OES chemistry results for Ti can be seen on Figure 5.

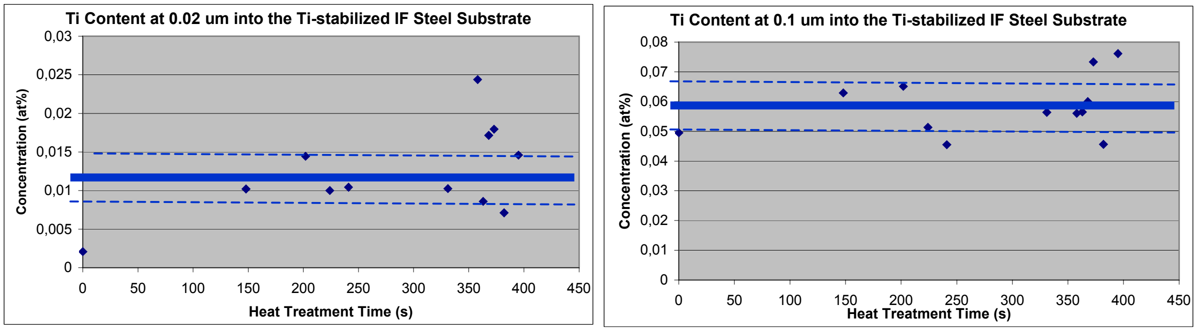


Figure 5. Ti surface concentration results along the thermal cycle set up for the Ti-stabilized IF steel grade (Ti bulk concentration is 0.062%).

On the other hand, a strong Nb enrichment was found at the nearest surface on both Ti- and dual-stabilized IF steel substrates. As soon as the samples started to be heated upwards the annealing peak, part of Nb in solid solution starts to diffuse towards the outermost surface of the steel substrate, driven by the tendency of chemical potential uniformity for this element between the bulk of the steel substrate and the surface. In the wake of the annealing heat treatment, those high Nb concentrations remain at the nearest surface all the way through the end of the GA heat treatment cycle. Besides, during galvannealing heat treatment no remarkable effect has been observed upon the Nb surface segregation. Nb surface concentration profiles are featured on Figure 6.

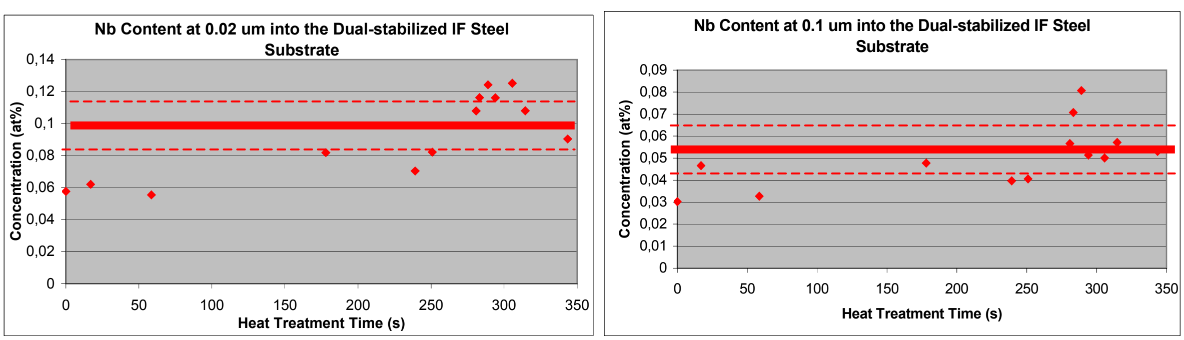


Figure 6. Nb surface concentration results along the thermal cycle set up for the dual-stabilized IF steel grade (Nb bulk concentration is 0.029%).

XPS analysis were carried out aiming to assess the Nb chemical state for the as-galvannealed condition at the surface of IF steels. For this purpose, the samples chosen for such analysis were those taken at the threshold of the GA heat treatment, which represent the setting condition for the subsequent galvannealing heat treatment. The XPS results showed a characteristic Nb $3d_{3/2}$ peak, which corresponds to Nb oxides in the form of Nb_2O_5, which can be seen on Figure 7.

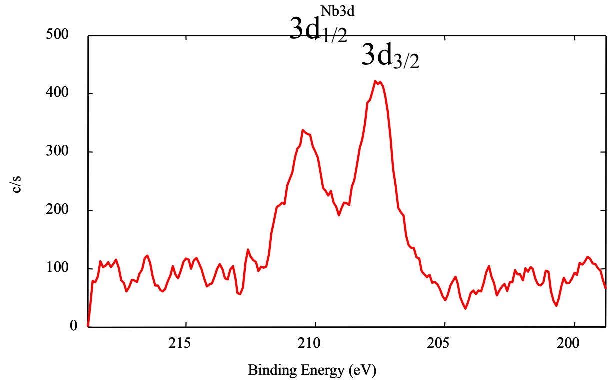
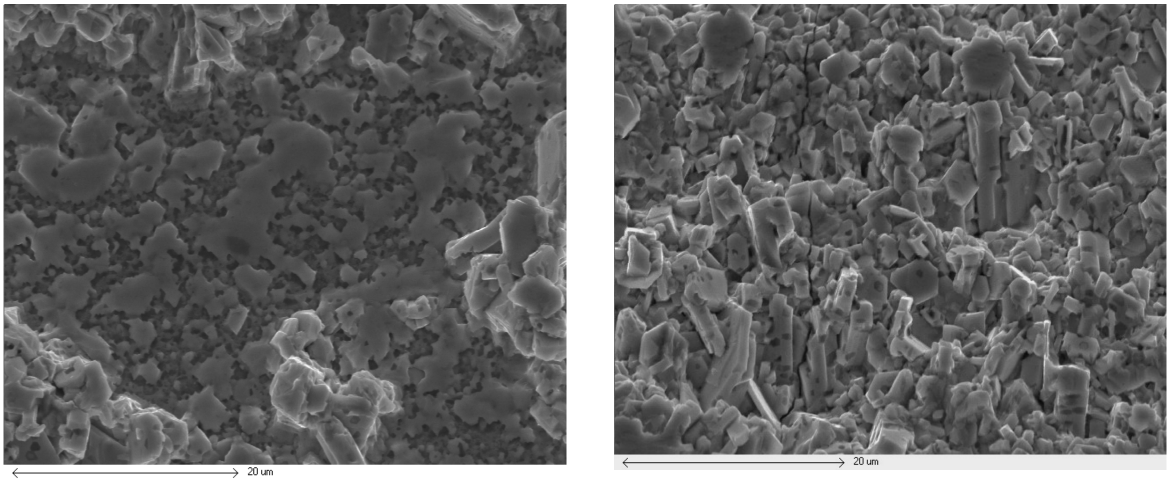


Figure 7. XPS surface analysis results for the dual-stabilized IF steel grade, featuring a Nb $3d_{3/2}$ peak whose binding energy is 207.4 eV), which corresponds to Nb_2O_5.

Therefore, it has been concluded that one of the reasons why the Ti-stabilized IF steel substrate is more reactive than the dual-stabilized IF steel substrate is that there are Nb oxides at the Zn coating-steel substrate interface blocking the Fe-Zn interdiffusion along the ferrite grain boundaries on the dual-stabilized IF steel substrate, whose Nb content is much higher than that for the Ti-only-stabilized IF steel substrate.

Light spectroscope and optical microscope micrographs were taken from the samples with streaks on top of GA coating. Two distinct types of streaks were found, a dark and a light one, which owe their distinctive features to differences in reflectivity and chemistry non-uniformity on top of the GA coating.

After that, SEM image analysis was used to magnify the field on dark and light streaked areas, and craters were found on the bottom of these regions. The areas featuring dark streaked areas showed a higher crater coverage when compared with the light streaked regions. EDS chemistry measurements were then carried out and showed that δ crystals were found on the bottom of craters on light streaked areas, whereas there is Γ layer on the bottom of craters on dark streaked areas. Therefore, the craters on dark streaked regions are deeper than those on light streaked areas. Micrographs and typical chemistries found for both kinds of craters are shown on Figure 8.



Element	wt%	at%
Al	1.91	4.35
Fe	21.39	23.54
Zn	76.70	72.11

Element	wt%	at%
Al	0.60	1.41
Fe	11.39	12.97
Zn	88.02	85.62

Figure 8. SEM images at 2300x featuring typical microstructures for a dark streaked area (left-hand side), whose EDS chemistry taken from the bottom matches a Γ phase, and light streaked area (right-hand side), whose chemistry is typical of a δ phase.

Regarding the Ti- and dual-stabilized IF steel panels that run interrupted thermal cycles under special cooling conditions at the HDP simulator in order to reproduce the operating conditions for crater formation in a CGL, further SEM analysis and EDS chemistry results showed that craters were only found on top of GA coatings for Ti-stabilized IF steel substrates, no matter if the panels were heat treated for 0s, 2s or 4s. On the other hand, no craters were found for dual-stabilized IF steel samples, regardless of the GA heat treatment time. Therefore, contending the previous work by most part of authors, Ti-stabilized IF steel substrates are more prone to crater formation than dual-stabilized IF steel substrates. Figure 9 shows the overall GA coating surface for the panels run for both IF steel grades.

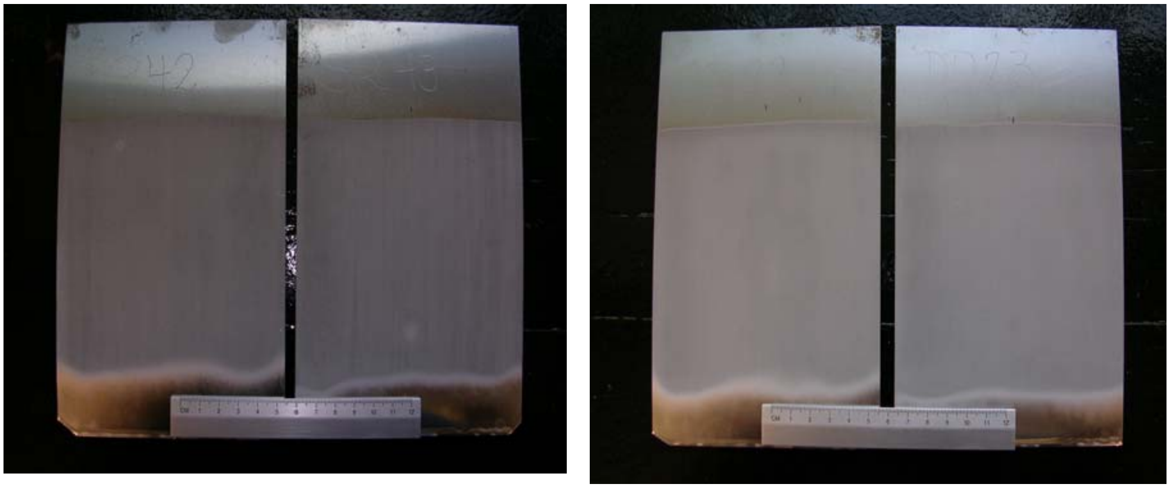


Figure 9. Pictures feature the overall outlook of Ti- (left-hand side) and dual-stabilized (right-hand side) IF steel panels that run interrupted GA thermal cycles under faster cooling rates.

5 CONCLUSIONS AND RECOMMENDATIONS

1. Ti depletion was found at the outermost surface on both IF steel grades, whereas this region features a strong Nb enrichment;
2. According to XPS analysis, this Nb surface enrichment corresponds to Nb oxides (Nb_2O_5). The presence of Nb oxides at the surface of dual-stabilized IF steel substrates blocks the Fe-Zn interdiffusion along the ferrite grain boundaries;
3. Simulations under special cooling conditions at the Hot-Dip Process Simulator found streaks only on Ti-stabilized IF steel substrate;
4. There is a higher number of craters on dark streaked areas when compared with the craters on light streaked areas;
5. δ crystals were found on the bottom of craters on light streaked areas, whereas there is Γ phase on the bottom of craters on dark streaked areas. Therefore, the craters on light streaked areas are shallower than those on dark streaked areas.
6. Due to the presence of Nb oxides at the interface between the Zn coating and the dual-stabilized IF steel substrate, the Ti-only-stabilized IF steel substrate is more reactive and consequently more prone than the dual-stabilized IF steel substrate to the formation of outbursts, and consequently craters.

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