Effect of Finish Hot Rolling Temperature on the Surface Quality of Galvannealed IF-HS Steel Sheets

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1. Introduction

Galvannealed IF-HS steels are currently the subject of immense interest for automotive industry for high corrosion resistance, considerably high strength with excellent formability and offer a huge potential for weight savings in car body. Many kinds of defects coming from the upstream processes, prior to and during galvanizing can lead to the deterioration of the surface quality of the coated sheet. During the heating and hot working of steel, the surface layer of oxide formed consists of three oxides: adjacent to metal surface is wustite (FeO), the intermediate layer of magnetite (Fe3O4) and the top layer of hematite (Fe2O3). The rate of oxidation is dependent on the temperature, composition and the physical characteristics of the steel, as well as the time of exposure of the metal surface to the oxidising atmosphere. Above 900°C, the hematite and magnetite content in the scale increases rapidly; whereas, around 850°C, the hematite content in the oxide is least and the wustite content is maximum. The thickness of scale remaining on hot rolled coils is mainly governed by the finish rolling temperature (FRT). Regardless of coiling temperature (CT), high FRT invariably results in greater amount of oxide scale on steel surface. Sticky, thin oxide scale formed during hot rolling may not get completely pickled out and subsequently give rise to the formation of a common surface defect called “bare-spot”, particularly on galvannealed IF-HS steel sheets. High strength steels containing higher amount of Mn and Si can segregate on the steel surface and oxidize during continuous annealing operation. Bare spots in galvanised steel are also reported to be associated with excess surface oxidation of these alloying elements. Dew point of IF-HS steel are also reported to be associated with excess surface oxidation of these alloying elements. Dew point of IF-HS steel are also reported to be associated with excess surface oxidation of these alloying elements.

2. Experimental Procedure

Galvannealed IF-HS samples containing bare spots were taken for experimental study. Bare spots are the areas not coated by liquid zinc on the surface of galvannealed steel sheets. The composition of the IF-HS steel, obtained by using an Inductively Coupled Plasma (ICP) spectrometer is given in Table 1. These steels are Mn and P strengthened, Ti-Nb stabilized IF-HS grade. Phosphorus is used in order to increase the strength of IF steel but is limited by cold work embrittlement (CWE) to ~0.08 wt%. Higher strength is obtained by judicious addition of Mn. The properties of galvannealed IF and galvannealed high strength re-phosphorised IF steels (IF-HS-GA) are shown in Table 2.

Uncoated spots were present on both top and bottom surface and are shown in the Fig. 1. Scanning Electron Microscopy (SEM) along with Energy Dispersive Spectroscopy (EDS) analysis using JXA-6400 was carried out on the uncoated spots. Point-wise EDS analysis was carried out starting from the centre of the defect up to the zinc coated area. An IF-HS (GA) sheet containing a relatively large bare spot (~4 mm diameter) was selected for X-ray Diffraction (XRD) analysis and a circular beam diameter was adjusted to focus on the bare spot only. The analysis was carried out using Co(Kα) radiation (λCo(Kα1)=1.788965 Å and λCo(Kα2)=1.792850 Å) to investigate the nature of compound present at the defect area. Quantitative Depth Profiling (QDP) was also carried out using Auger Electron Spectroscopy (AES) with an acceleration voltage of 10.0 kV, current (Ip) of 4.31×10^-6 A and sputter rate of 100 Å/min in order to determine the depth profile of the defects. The spectroscopy curves from the surface, at 300 Å, 600 Å, 0.16 µm, 0.365 µm and 0.415 µm depths were obtained and analyzed.

3. Results and Discussion

The uncoated spots were brown in colour, random in nature, slightly elongated in the rolling direction and generally 0.5–2.0 mm (major axis) in size with an aspect ratio of 2.3–4.0. These spots appeared in single or in cluster of small islands (Fig. 1). Figure 2 shows the SEM image of the defect area along with the points of EDS analysis. Strong peaks of Fe were observed at the center of the defect (Fig. 3), whereas, Zn and Fe peaks were observed at the perfectly galvannealed area, as expected (Fig. 4). Absence of Zn and presence of Fe at the defect area was confirmed from the elemental mapping in SEM-EDS. In X-ray diffraction study, three major peaks of Fe along with the peaks of Fe2O3, Fe3O4, FeO, FeFe2O4, Fe0.98O, Fe3P, Al and Zn were detected (Table 3). The surface analysis of the bare spot

### Table 1. Chemical composition of IF-HS steel.

|   | C | Mn | S | P | Si | Al | Cu | Cr | Ni | Mo | V | Nb | Ti | B |
|---|---|----|---|---|----|----|----|----|----|----|---|----|----|---|
|   | 0.0035 | 0.38 | 0.006 | 0.044 | 0.013 | 0.040 | 0.005 | 0.044 | 0.012 | <0.005 | <0.005 | 0.040 | 0.038 | 0.0008 |

### Table 2. Properties of IF(GA) and IF-HS (GA).

| Steel | Y.S. (MPa) | U.T.S. (MPa) | Elongation (%) | t-bar |
|-------|------------|--------------|----------------|-------|
| IF (GA) | 180 | 330 | 38 | 1.6 |
| IF-HS (GA) | 240 | 450 | 38 | 1.4 |
and compositional variation along the depth was investigated by AES. The intensity of the oxygen peak decreased along the depth of the defect. The gradual depletion of oxygen level signifies the presence of complex oxide scale on the defect area. The AES profiles at the surface of the defect and at 0.415 mm depth are shown in Figs. 5(a) and 5(b).

The problem of uncoated spots and dewettability or non-adherence of zinc coating on particular defect areas for high strength interstitial free steel can be explained in two ways: possible peeling of zinc after galvanising and/or the presence of oxides or other compounds at that particular defect area, which is not wettable by liquid zinc. The X-ray diffraction study showed the presence of complex iron oxide comprising of FeO, Fe₂O₃, Fe₃O₄, Fe₀.⁹₈O and FeFe₂O₄ at the defect spot. It is therefore important to know about the processing stages that can form these complex oxides. They can be formed in two stages during the processing of steel: firstly, inside the continuous annealing furnace of the continuous galvanising line and secondly, dur-

| Element/Compound | d(Å)         |
|------------------|--------------|
| Fe               | 2.02, 1.43, 1.17 |
| Fe₂O₃            | 1.92, 2.36, 2.51, 2.08, 2.40, 1.81, 1.27 |
| Fe₃O₄            | 2.52, 2.53, 2.42, 2.41 |
| FeO               | 2.04, 2.59, 1.15 |
| FeFe₂O₄          | 2.60, 2.03, 1.23, 1.90 |
| Fe₃O₄            | 2.08, 2.52, 2.51, 2.37, 2.07, 1.51, 1.44, 1.29 |
| Al                | 2.34, 2.02, 1.43, 1.22, 1.17 |
| Zn                | 2.413, 2.29, 2.08, 1.33, 1.24, 1.17 |
ing hot rolling of steel. From the visual inspection of defects, it was found that these defects were oriented in the rolled-in direction. A particular IF-HS coil was tracked from hot rolling stage up to the end of galvannealing. Plenty of red oxide scales embedded on the surface in the rolled-in direction was observed.

If we assume that the oxide forms in the continuous annealing furnace of hot dip galvanising line, it should form homogeneously, and should not have any preferential site of formation. It should not have any rolled-in appearance, as no major deformation is given after galvanising. It is very much unlikely to form iron oxide inside an annealing furnace, which is maintained at reducing atmosphere of 96%N₂-4%H₂. The possibility of formation of iron oxide inside the continuous annealing furnace is thus comparatively less.

Oxides containing higher amount of Fe₂O₃ or Fe₃O₄ at the top of the scale formed during finish hot rolling are difficult to dissolve in HCl at the time of pickling. The unpickled or partially picked oxides may get rolled in during cold deformation and subsequently give dewettability problem during hot dip galvanising. Therefore, a thin scale having maximum amount of FeO and least Fe₃O₄ is considered to have good pickling characteristics. It has been observed that IF-HS steels containing higher amount of P (~0.04%) has a non-uniform picklability in the tandem pickling line and cold rolling mill. Certain metals like Cu, Cr and Ni retard the rate of pickling when these elements occur in the steel base. Si and Al also retard the solubility rate of oxide in the acid.

During the heating and hot working of steel, the surface layer of oxide formed is known as mill scale and consists of three oxides: ferrous oxide or wustite (FeO), magnetite or ferrous-ferric oxide (Fe₂O₃) and ferric oxide or hematite (Fe₃O₄). Ferrous oxide, the layer next to the metal surface constitutes about 85% of the scale thickness, the magnetite about 10–15% and the ferric oxide about 0.5–2%. According to Garber and Sturgen, the mole fraction of iron oxide in the mill scale changes with temperature; above 900°C, the mole fraction of magnetite and hematite in scale increases and that of wustite decreases.

In general, hot rolled strips with higher finish rolling temperature have thicker scale on their surface, which is not fractured and adheres well to the steel substrate, requiring more acid and longer time for proper pickling. It was observed in this study that the finish rolling temperature for Ti-Nb stabilised IF-HS steels were maintained in the range of 905 to 940°C (mostly >920°C). This produces sticky high temperature oxides that cannot be properly pickled in the picking line giving rise to the dewettability problem during hot dip galvanising.

Based on the experimental studies, observations and theoretical evidence, it was recommended to maintain the finish rolling temperature in hot rolling of this IF-HS grade close to 860°C. This would generate the least amount of hematite (Fe₂O₃) and magnetite (Fe₃O₄) in the scale. This would also ensure maximum amount of wustite (FeO) in scale that will have better pickling property. Thus the problem of dewettability and bare spot during hot dip galvanising can be eliminated. On the basis of this recommendation, a plant trial has been carried out for a single coil rolled at FRT <900°C (Fig. 6). In order to roll the IF-HS slabs at low finish rolling temperature of ~900°C, the slab drop out temperature from hot rolling mill reheating furnace was fixed at 1190°C, keeping in mind that the mill load at any stage does not exceed the critical limit. The result is encouraging; the coil ends up with no uncoated spot after galvannealing.

4. Conclusion

The systematic surface characterization study on galvannealed IF-HS steel sheets revealed that a high FRT (930–940°C) could lead to the formation of sticky high temperature oxides (Fe₂O₃ and Fe₃O₄) on hot rolled coils, which are very difficult to be pickled out completely. The left-over tiny oxide scales get rolled in during cold deformation and are the prime cause of uncoated spots after hot dip galvanizing. Hot rolling at a lower FRT (~880°C) could minimize the formation of such oxide scales and thus improve the surface quality of galvannealed IF-HS steel sheets with no uncoated spots.

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