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## Development of the surface structure of TRIP steels prior to hot-dip galvanizing

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#### Abstract

Focusing on improving the reactive wetting of transformation-induced plasticity (TRIP) steels during hot-dip galvanizing, the effect of the alloying elements manganese, silicon and aluminum on the surface structure prior to galvanizing was studied. A C–Mn steel and two TRIP steels with aluminum either completely or partially replacing silicon were investigated. The two TRIP steels showed good wettability when using a -30 °C dew point with a 95% N<sub>2</sub>–5% H<sub>2</sub> atmosphere and improved wettability when using a -53 °C dew point with a 80% N<sub>2</sub>–20% H<sub>2</sub> atmosphere. The decreased oxide thickness and the change in the distribution of oxides on the surface, showing a larger surface area that was free of oxide particles, contributed to the improved reactive wetting of the two TRIP steels at the -53 °C dew point. The C–Mn steel demonstrated excellent wettability for both annealing atmospheres. The observed reactive wetting was attributed to the reduction of MnO by Al in the Zn(Al, Fe) bath. © 2006 Elsevier B.V. All rights reserved.

Keywords: Galvanizing; Transformation-induced plasticity steel; Gas-metal reactions; Selective oxidation

### 1. Introduction

Transformation-induced plasticity assisted steels (TRIP steels) are ideal candidates for lightweight automotive applications due to their high strength to weight ratio, allowing thinner cross-sections to be used for a given application [1]. Furthermore, the good uniform elongation and high work hardening rate of TRIP steels provide high strength without compromising ductility, formability and vehicle crashworthiness [1]. However, the use of thinner cross-sections results in corrosion protection becoming increasingly important in order to maintain structural integrity and satisfy consumer durability expectations. The continuous galvanizing process is among the most cost-effective means of achieving this objective, but when using conventional hot-dip galvanizing process parameters there can be problems with poor reactive wetting between the liquid zinc and the surface of TRIP steels [2,3], which is necessary for the formation of the interfacial inhibition layer (Fe<sub>2</sub>Al<sub>5</sub>) and is integral to producing coatings with high ductility [4]. Poor wetting is usually caused by the selective oxidation of the Mn, Si and Al that are

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commonly used as alloying elements in these steels, i.e., they form oxides that are not reduced by conventional atmospheres employed in continuous galvanizing.

TRIP steels have conventionally contained C, Mn and Si [5], where C and Mn are used to stabilize austenite and Si is used as a solid solution strengthener and to suppress carbide formation [6,7]. Silicon often causes problems during hot rolling, producing an oxide scale that is difficult to remove in subsequent processing [8,9]. Silicon has also been shown to be problematic during hot-dip galvanizing [2], causing poor wettability as a result of a layer of amorphous SiO<sub>2</sub> present on the surface [10]. It has also been shown that Mn and Si can form the mixed oxide Mn<sub>2</sub>SiO<sub>4</sub>, which was also found to be detrimental to the wettability of the steel [11]. Due to the adverse affects of Si during continuous galvanizing, Al has been selectively substituted for Si in TRIP steels and has been shown to improve reactive wetting during galvanizing [11]. Al has also been shown to have a similar effect to Si on TRIP steel microstructural evolution, preventing carbide precipitation and providing a sufficient amount of retained austenite that is stable at room temperature [12]. De Meyer et al. [8] have shown that Al can partially replace Si to produce TRIP steels with comparable strength and superior ductility to those with a higher Si content. In this study, TRIP steels alloyed with C, Mn and with Al either completely or partially

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Table 1	
Chemical composition of steels	(wt.%)

Alloy name	С	Mn	Al	Si	Ti	Cu	Cr	Nb	O [ppm]	N [ppm]
1.5% AI TRIP	0.20	1.55	1.52	0.025	0.025	n/a	n/a	n/a	1	20
1% AI TRIP C–Mn	0.23	1.52 1.4	1.05 n/a	0.55 0.09	0.026	n/a 0.018	n/a 0.012	n/a 0.071	4 n/a	60

replacing Si have been used for the purpose of investigating the reactive wetting of TRIP steels during continuous galvanizing.

In this investigation, the galvanizing of two TRIP steels and one C–Mn steel was studied. The wettability of the alloys by liquid Zn(Al, Fe) was investigated (i) by the visual appearance of the galvanized panels and (ii) by observing the inhibition layer (Fe<sub>2</sub>Al<sub>5</sub>) at the Fe–Zn interface. The wettability of the steel will be related to the surface structure prior to galvanizing, focusing on the selective oxidation of alloying elements at the surface. The depth to which the oxides penetrate, the chemical composition of the oxides and the distribution of oxides on the surface was studied. The steel surface morphology was also observed at various times during the annealing cycle to determine how the surface structure evolved with annealing time.

### 2. Experimental method

The chemical compositions of the experimental steels are shown in Table 1. The TRIP steels were fabricated at the CAN-MET Materials Technology Laboratory and the C–Mn steel was supplied by Stelco Inc.

Experimental heat treatment consisted of heating the steel to the intercritical annealing temperature at which the microstructure would be 50%  $\alpha$ -50%  $\gamma$ . This temperature was determined using Thermocalc<sup>®</sup> to be 862 °C for the 1.5% Al TRIP steel and 825 °C for the 1% Al TRIP steel. The C-Mn steel was tested at both intercritical annealing temperatures and these samples will be distinguished in the subsequent text by the annealing temperature (i.e., 'C-Mn 825 °C' and 'C-Mn 862 °C'). The steel was held at the intercritical annealing temperature for 120 s, cooled at  $20 \,^{\circ}\text{C}\,\text{s}^{-1}$  to  $460 \,^{\circ}\text{C}$  and immersed in the Zn(Al, Fe) bath for 4 s. Samples that were not dipped were held isothermally at 460 °C for 4 s. As an illustration, the heat treatment used for the 1.5% Al TRIP steel is shown in Fig. 1. For samples that were annealed without galvanizing, the experiment was interrupted at three different points during the heat treatment by quenching the sample to room temperature, as shown in Fig. 1.

All experiments were carried out in the McMaster Galvanizing Simulator (MGS). The MGS consists of an atmosphere controlled column composed of several sections (described from top to bottom of the apparatus) (i) sample loading/cooling, (ii) sample heating, (iii) gas jet wiping and (iv) zinc pot. The sample loading/cooling section contains two parallel gas cooling platens. The heating section uses two furnaces: (i) a quartz lamp infrared (IR) furnace and (ii) a high frequency (HF) furnace; only the former was used in this work. The two upper sections are separated from the gas jet wiping and zinc pot by an airlock. Gas jet wiping controls the coating weight using a N<sub>2</sub> gas jet yielding an average coating weight of 69 g m<sup>-2</sup> for the galvanized sam-



Fig. 1. Heat treatment profile for 1.5% Al TRIP steel, showing sampling points for bare panels.

ples. The zinc pot consists of a resistance heated 50 kg graphite crucible controlled to within  $\pm 2$  °C using a conventional process controller. The zinc bath contained 0.20 wt.% dissolved Al and was iron saturated [13,14]. The sample heating/cooling thermal cycle was controlled using a type K (0.5 mm) thermocouple welded directly to the sample prior to the start of the experiment.

The steel panels used in the galvanizing simulator were  $120 \text{ mm} \times 200 \text{ mm}$  with the longitudinal axis of the sample parallel to the rolling direction. Samples were approximately 1.5 mm thick. A uniform temperature and coating area on the panel of  $90 \text{ mm} \times 90 \text{ mm}$  is centered in the lower portion of the steel panel and only material from this area was analyzed. Prior to annealing or galvanizing the steel panels were cleaned in a 2% NaOH solution, rinsed with water, cleaned ultrasonically in isopropanol and dried with warm air. The samples that were annealed without galvanizing were also polished to a mirror finish to minimize the effects of surface roughness on the subsequent surface analysis. A final cleaning with acetone was performed immediately prior to the sample entering the galvanizing simulator. To protect the annealed panels from further oxidation, samples were stored in isopropanol immediately after being removed from the MGS.

Two reducing atmospheres were tested and their compositions are summarized in Table 2. The  $P_{\text{H}_2\text{O}}/P_{\text{H}_2}$  ratio was

 Table 2

 Experimental atmospheres

Atmosphere	Dew point [°C]	Nitrogen content [vol.%]	Hydrogen content [vol.%]	$P_{\mathrm{H}_{2}\mathrm{O}}/P_{\mathrm{H}_{2}}$
−30 °C DP	-30	95	5	0.00844
−53 °C DP	-53	80	20	0.000191

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