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## Relation between emissivity evolution during annealing and selective oxidation of TRIP steel

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### ABSTRACT

Spectral emissivity measurement ( $\lambda = 8\text{--}12\ \mu\text{m}$ ) of TRIP alloy steel during annealing in  $\text{HN}_x$  atmosphere is carried out. Emissivity variation is contributed by the selective oxidation that takes place along with the surface topography from pre-annealed state. Comparing results from physicochemical characterization (Fourier transform infrared spectroscopy, scanning electron microscopy and optical surface profiler) on post-annealed samples and examining the global and relative spectral emissivity separately established correlation of roughness to the former and oxide signatures to the peak features in the latter. Intensity of emissivity peaks at  $10\ \mu\text{m}$  and  $10.5\ \mu\text{m}$  vary with ratio of Mn to Al. Meanwhile, the influence of local texture on emissivity is demonstrated.

### 1. Introduction

The ever rising requirements for steel products in the automotive industry, which include weight reduction, improved mechanical properties for safety measures and manufacturing processes, have led to the production of high performance steels such as transformation induced plasticity (TRIP) and dual phase (DP). Compared to conventional steel, higher percentage of alloying elements (Mn, Al, Si) are present in these steel grades. During annealing, the more reactive alloying elements will segregate from the bulk towards the surface and form selective oxides that are detrimental for coating adherence during galvanization. Hence, the study of selective oxides is crucial for quality control purpose.

The formation of selective oxides has been investigated extensively in [1–9]. The classical Wagner model gives the critical mole fraction of the alloy element in a single crystal binary alloy, where the inward flow of oxygen is blocked and external oxidation takes place. Below this critical mole fraction, internal oxidation is expected [1,6,10]. Maigne et al. [8] considered grain boundary oxidation by multiplying the activation energy with a factor of 0.5 and put forward an inequality taking into consideration the sum of all oxides as a criterion for the transition to external oxidation. This model does not take into account site competition of segregating solutes reported by Grabke et al. [3] and treat the oxidation of different species separately. By extending the classical internal and external oxidation model after considering permeability of oxygen and solute elements as well as solubility-precipitation, Lashgari [6] developed more detailed models for binary

alloy Fe-M, setting criteria for cases where internal oxidation and formation of external scale co-exist and where they occur individually. Simultaneous diffusion and precipitation of chemical elements are considered by models proposed by Huin et al. [2,4] along with numerical implementations.

Besides the influence of temperature, the formation of the different species of selective oxides is also governed by the diffusion of oxygen and alloying elements that result in concentration variations across the depth. As the subsurface depth increases, the concentration of dissolved oxygen decreases. Kim et al. [5] reported that the formation of  $\text{Al}_2\text{O}_3$ ,  $\text{MnAl}_2\text{O}_4$  and MnO take place in an order of increasing partial pressure of oxygen. Liu et al. [7] studied the microstructure of annealed TRIP steel in the dew point range of  $-30$  to  $+10\ ^\circ\text{C}$  and found that at high dew point, decarburization and consumption of alloy elements leads to formation of larger subsurface ferrite. This in turn affects the segregation of alloying elements due to the lower diffusivity of Al in ferrite than Mn [11]. Generally, oxidation takes place in a discontinuous manner and unstable mixed oxide formation is reversible.

The morphology of selective oxides on the surface varies with steel chemistry and dew point. Grainy oxides (islands) are found on the surface of TRIP steel annealed at high dew point in contrast to the film-like oxides at low dew point [12–14]. Sagl et al. [12] also reported increased crystallinity with increased dew point. It could hence be summarized that selective oxidation that takes place depending on annealing conditions alters the surface topography and chemical composition, resulting in heterogeneous distribution.

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The change in the surface oxidation state results in emissivity variation [15–18]. Based on this *a priori* knowledge, the study aims to explore the indirect method of inferring oxidation state of the chosen TRIP steel grade from in-situ emissivity measurement carried out during the annealing. For this purpose, a set-up in the laboratory to simulate the annealing thermal cycle which enables simultaneous normal emissivity measurement is employed. To the best of the authors' knowledge, this is the first time that in-situ spectral emissivity (8–12  $\mu\text{m}$ ) is reported for selective oxidation-prone steel grade.

Since the alloy steel is heated to elevated temperatures ( $> 800\text{ }^\circ\text{C}$ ) and the chemical potential changes due to the diffusion of oxygen from the atmosphere and alloying elements from the bulk, the overall radiative properties during annealing vary according to temperature-dependent properties (e.g. resistance) and chemical composition-related properties [19–21]. Moreover, studies have demonstrated that the surface topography and microstructure also contribute to the resultant radiance observed [22–25].

This paper investigates the aspects mentioned above and looks into the contribution of these factors at different length scales to the macroscopic property manifested by the emissivity.

## 2. Experiments

### 2.1. Set-up

Sheets of cold-rolled TRIP (nominal composition 1.74 wt.% of Mn, 1.41 wt.% of Al and 0.08 wt.% of Si) with 1 mm thickness are cut to 15 cm  $\times$  2 cm. For reference, cold-rolled interstitial free (IF) standard steel is also applied. Prior to annealing, black paint ( $\epsilon_{\lambda} = 0.8\text{--}1.6\text{ }\mu\text{m} \sim 0.93$  up till  $900\text{ }^\circ\text{C}$ ) is applied on one side of the sample and dried subsequently. This black-painted side with known emissivity is used for temperature regulation by a pyrometer ( $\lambda = 0.8\text{--}1.6\text{ }\mu\text{m}$ ) during the annealing thermal cycle. As illustrated in Fig. 1, the annealing apparatus consists of an enclosed black cavity with quartz window on one side, transparent in the wavelength range of the regulation pyrometer. On the other side, a ZnSe window with anti-reflection coating is employed, which transmits  $> 90\%$  in the infrared region. The normal emissivity in the long wavelength range (LWIR), 8–12  $\mu\text{m}$  is obtained by a bolometric spectrometer. The black cavity is purged with  $\text{HN}_x$  gas (a mixture of  $\text{N}_2$  and 4.5% of  $\text{H}_2$ ) and the atmosphere inside the cavity is monitored by a hygrometer and an  $\text{O}_2$  analyzer. Both ends of the sample are fixed with a set of clamping jaws inside the cavity where the sample is heated by means of Joule effect. The temperature distribution was verified by an infrared camera and a homogeneous zone of  $2 \times 1\text{ cm}$  with a temperature variation inferior

to  $\pm 5\text{ }^\circ\text{C}$  at  $800\text{ }^\circ\text{C}$  was observed. The selected area that was taken into account to obtain the emissivity was inferior to this and remains within the corresponding pyrometer regulated spot ( $\sim 1\text{ cm}$ ). The PID controller reads the real-time temperature measured by the regulated pyrometer to generate the current for heating in order to adhere to the pre-programmed thermal cycle. As background information, this laboratory annealing set-up is designed to study the thermal cycle that the steel is subjected to in the production line. Therefore the thermal cycle programmed here aims to represent the industrial thermal treatment of the TRIP steel grade. However, due to different source of heating, temperature and atmosphere control, discrepancies are anticipated between laboratory and industrial thermal treatment.

### 2.2. Experimental variables

The desired dew point (DP) of the atmosphere is attained by adjusting the mixture of the flow rates of humidified  $\text{HN}_x$  and dry  $\text{HN}_x$ . The  $\text{O}_2$  concentration in the cavity is inferior to 5 ppm before the thermal cycle begins. In the first set of experiments, the samples are heated at  $2.2\text{ }^\circ\text{C/s}$  to  $860\text{ }^\circ\text{C}$  where they are maintained for a soaking time of 129 s under atmospheres of different dew points ( $0\text{ }^\circ\text{C}$ ,  $-6\text{ }^\circ\text{C}$ ,  $-10\text{ }^\circ\text{C}$  and  $-16\text{ }^\circ\text{C}$ ). In the second set, the soaking time ( $t_s$ ) is varied (60 s and 180 s) while the dew point is set at  $-6\text{ }^\circ\text{C}$  with the same heating rate ( $2.2\text{ }^\circ\text{C/s}$ ) and soaking temperature ( $860\text{ }^\circ\text{C}$ ). Each run is repeated at least twice and the representative emissivity values are presented in the following section. In addition, a polished sample ( $R_a = 15\text{ nm}$ ) is employed during the thermal cycle with DP =  $-6\text{ }^\circ\text{C}$  and  $t_s = 129\text{ s}$ . Chemical analysis of an as-received non-polished (NP) and a polished (P) sample by means of secondary ion mass spectrometry up to  $5\text{ }\mu\text{m}$  below the surface suggests no difference in the chemical composition distribution before annealing.

### 2.3. Measurements and data processing

During the annealing, real-time temperatures are measured by the pyrometer and the emitted radiance of the sample,  $S_{\lambda_n}$  is registered by a long wave infrared (LWIR) spectrometer normal to the sample surface. Within the range of 8–12  $\mu\text{m}$ , the spectrometer has 30 spectral bands. A collection area corresponding to the isothermal zone is evaluated. The acquisition frequency is 5 Hz. The properties of the black cavity prevent parasitic flux due to reflection of the atmosphere. By convention,

$$S_{\lambda_n} = \epsilon \cdot B_{\lambda_n} \quad B_{\lambda_n} = t(\lambda)k(\lambda) \cdot L_B \quad (1)$$

The emissivity of a non-blackbody is represented by  $\epsilon$ .  $L_B$  is the Planck's blackbody radiance. The blackbody signal,  $B_{\lambda_n}$  is computed

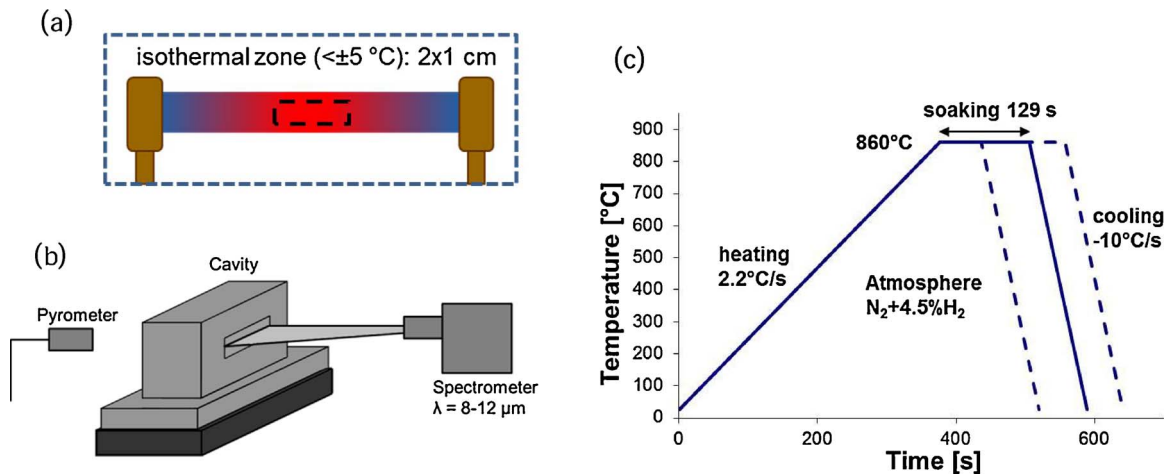


Fig. 1. Experiment set-up: (a) sample in the cavity heated by Joule effect and analyzed isothermal zone, (b) instrument set-up (regulation pyrometer and LWIR spectrometer), (c) the thermal cycle of dew point variation series (full line) and soaking time variation series (dashed line).

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