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Heating rate effect on liquid Zn-assisted embrittlement of high Mn austenitic steel

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ABSTRACT

The effect of heating rate on liquid metal embrittlement (LME) of a galvanized twinning induced plasticity (TWIP) steel was investigated by hot tensile testing. The specimens were heated with different heating rates to 600, 700 and 800 °C, and elongated to 40%. During the hot tensile tests, various Fe-Zn intermetallic compounds were formed depending on the heating rate and the deformation temperature, and they were distinguished by X-ray diffraction and energy dispersive spectroscopy. δ and Γ were observed in the coating after 600 °C deformation regardless of heating rate. Since both phases did not liquefy at 600 °C, the steel was not embrittled. Heating to 700 °C sometimes produced δ which would be liquid at 700 °C. It was only produced at the highest heating rate and embrittled the steel. Γ was produced at 800 °C regardless of heating rate. Hence, the steel suffered from LME in all heating rate condition. Based on the results, the importance of selecting temperature and heating rate to study LME by hot deformation testing is discussed.

1. Introduction

The demands for advanced high strength steels (AHSS) have recently been increased to produce lightweight automotive body and increase passenger safety simultaneously [1]. The AHSS are generally coated with Zn because of its low cost, sacrificial protection and good surface quality [2]. In addition, the weldability of these steels has to be guaranteed to assemble steel components into a car body [3]. Since resistance spot welding (RSW) is fast and economical, it is the most widely used joining method in the automotive industry. However, some galvanized AHSS are degraded by cracks due to liquid metal embrittlement (LME) after the RSW process [4,5]. LME is the degradation of ductility of the alloys under tensile stress, while the alloys are in contact with liquid metals [6]. During RSW of the Zn coated steels, Zn melts during the heating, because its melting point is much lower than that of the steel substrate, and some parts of the sheet steel undergo tensile stress. Therefore, LME occurs during RSW of Zn coated sheet steels, and the produced cracks decrease the mechanical reliability of the welds. Recently, LME during RSW has been an issue especially in twinning induced plasticity (TWIP) steels in which the cracks larger than 200 μm appear [3–5]. Although TWIP steel has superior strength and elongation, the LME cracks generated during RSW limit the applicability of the TWIP steel to the automotive industry.

When the sheet steels are welded by RSW, each part of the sheet steel experiences different thermal and mechanical history. In addition, the normal welding time is < 2 s; hence, it is hard to systematically analyze the LME during RSW. Since hot tensile tests can control main factors for LME, such as temperature, stress, strain rate, as well as heating and cooling rate, numerous studies on LME have used hot tensile testing [3,7–10]. The adopted conditions and observed phases are listed in Table 1. The studies in [3,9] tried to identify the temperature and strain rate condition at which LME occurred. The work in [8] was carried out to discuss the mechanism of LME in TWIP. The others [7,10] claimed that the microcracks in the substrate adjacent to coating after hot stamping of 22MnB5 steel was caused by LME and attempted to clarify the corresponding mechanism. The study of LME in [7,8,10] was limited to 800–950 °C at which hot stamping takes place. Γ was always observed when LME occurred, since Γ is liquid at 800–950 °C. Although [3,9] carried out testing at wide temperature range, the phases in the coatings were never observed. In all these studies, various heating rates from 2.5 to 80 °C s^{-1} were employed without any explanation. However, the temperature as well as heating rate can influence the formation of Fe-Zn intermetallic compounds. Higher heating rate can limit the interdiffusion time and the diffusion also strongly depends on the temperature. Since the melting points of these compounds increase with the Fe concentration, the probability of the contact between liquid Zn and a steel substrate is changed by the deformation temperature and heating rate.

The present study aims to emphasize the importance of selecting the test condition when hot deformation testing is carried out to investigate

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Table 1
Testing conditions and observed phases in the coating reported in [3,7–10].

Ref.	Alloy type	Heating rate/°C s^{-1}	Temperature/°C	Holding time/min	Strain rate/s^{-1}	Observed phases in the coating
[3]	TWIP steel	80	400–1000	≤5	1.3×10^{-3} - 1.3	Not mentioned
[7]	22MnB5	Not mentioned	900	0.5	Not mentioned	Γ + α-Fe(Zn)
[8]	TWIP steel	10	850	0.5	0.5	Γ + γ(Fe,Mn)(Zn)
	Ti-IF steel					Γ + α-Fe(Zn)
	Ti-IF steel	2.5	950	0.5	0.5	Γ + α-Fe(Zn)
[9]	DQ, DP, TWIP steels	20	600–900	1	10^{-2} - 1	Not mentioned
[10]	22MnB5	20	850	4–20	0.5	Γ + α-Fe(Zn) or α-Fe(Zn)

Table 2
Chemical composition of the TWIP steel. Fe is balanced.

C/wt%	Mn/wt%	Al/wt%	Cr/wt%	Si/wt%	P/wt%	S/wt%	N/wt%	O/wt%
0.46	16.4	1.5	0.3	0.1	0.008	0.001	0.005	0.002

LME. To achieve the aim, the present work studies the formation of Fe-Zn intermetallic compounds and its influence on LME by heating a galvanized TWIP steel under different heating rates and subsequently tensile straining the steel at 600, 700 and 800 °C.

2. Experimental procedures

Cold-rolled galvanized TWIP sheet steels were prepared for hot tensile tests and its composition is listed in Table 2. The steel sheets were 1.4 mm in thickness. A 100–200 nm-thick Ni layer was electroplated on the steel to ensure galvanizability, which prevented the formation of an inhibition layer between the coating and Fe substrate. The cold-rolled sheets were Zn coated in a continuous galvanizing line where the 0.12 wt%Al-Zn bath temperature was around 450 °C. The coating thickness ranged 7–10 μm. It was machined into dog bone-shape tensile specimens for the hot tensile tests, and the gauge width and length were 12.5 and 30 mm, respectively.

Hot tensile tests were conducted by a thermo-mechanical process simulator, Gleeble 3500. Firstly, the tensile specimens were cleaned with acetone to remove contaminants, and then thermocouples were welded to the center of the specimen to measure and control the temperature. The samples were heated with three different heating rates (4, 20, 100 °C s^{-1}) to 600, 700 and 800 °C in air. The deformation temperatures were selected considering the melting points of η (419 °C), δ (672 °C), and Γ (782 °C). Therefore, only η is liquid at 600 °C, both η and δ are liquid at 700 °C, and η, δ, and Γ are liquid at 800 °C. After 1 s of holding, the specimens were deformed to the engineering strain of 40% with a strain rate of 0.1 s^{-1}. Then, they were cooled down to room temperature at a cooling rate of 80 °C s^{-1} by cooling with compressive air. In case of the specimen heated up to 700 °C, additional experiments without deformation were conducted to observe a fine influence on Fe-Zn reaction by the high temperature exposure during the loading. The hot deformed specimens were classified into either fractured or intact. Since the fracture surface of the tensile specimen had been oxidized during hot tensile test, it could not provide any meaningful information about the phases involved in LME. Thus, the periphery of the fracture surface was cut in the case of the fractured specimens. The most severely deformed part was sectioned for the intact specimens.

To determine the existence of Fe-Zn intermetallic compounds in the coating, the sectioned specimens were cleaned with acetone, and X-Ray Diffraction (XRD) analysis was conducted by a Bruker XRD D8 Advance Davinci Diffractometer with a Cu target which yielded a wavelength equal to 0.154062 nm. The scan angle (2θ) range was from 35° to 55°. Since only few peaks for ZnO and α-Fe(Zn) appeared in the range, the results for higher 2θ were attached as a Supplementary material (Figs. S1–S5). The crystallographic information of η [11], ζ [12], δ [13], Γ [14], α (JCPDS 06-0696), and γ (JCPDS 33-0397) was obtained from the literatures and JCPDS cards.

The steel coupons were mounted in such an orientation to observe the plane normal to rolling direction. The mounted specimens were ground and polished from a 400 P sandpaper to colloidal silica. The polished specimen was observed by a JEOL JXM-7100F field emission scanning electron microscope (FE-SEM). The compositional data were obtained by an Oxford energy dispersive spectroscopy (EDS) detector under 20 kV. The back scattered electron (BSE) images were also taken under the same acceleration voltage to clarify the phase distinctions and the compositional differences.

3. Results

Hot tensile stress-strain curves of the galvanized TWIP steel are given in Fig. 1. Generally, the strength of a steel decreases and its elongation increases as the deformation temperature increases. The temperature dependence of the strength in Fig. 1 is coherent with such behavior, but the elongation was reduced at 700 and 800 °C. There was no fracture or ductility reduction in TWIP steels at 600 °C. Interestingly,

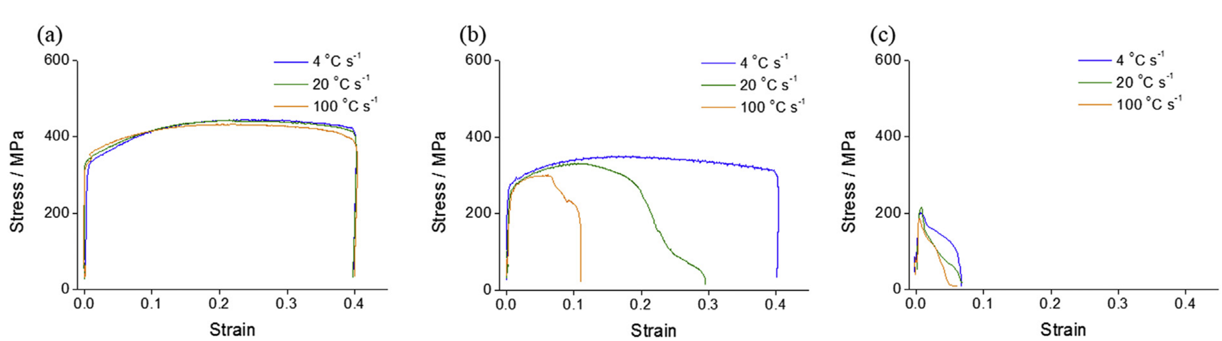

Fig. 1. Hot tensile test results of galvanized TWIP steel at (a) 600 °C, (b) 700 °C [15], (c) 800 °C with different heating rates.