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# Effects of intergranular carbide precipitation on delayed fracture behavior in three TWinning Induced Plasticity (TWIP) steels



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#### ABSTRACT

The delayed fracture behavior related with intergranular carbide precipitation of three TWinning Induced Plasticity (TWIP) steels was investigated. According to the microstructural analysis, nanosized (Fe,Mn)<sub>3</sub>C cementites were precipitated along grain boundaries in the 0.6C–22Mn and 0.6C–18Mn steels, whereas their precipitation was hardly observed in the 0.6C–18Mn–2Al steel, which was confirmed by equilibrium phase diagrams calculated from a ThermoCalc program. When cup specimens were dipped in the boiled water, the 0.6C–22Mn, 0.6C–18Mn, and 0.6C–18Mn–2Al steel cups were cracked after 5.5, 15, and 169 h, respectively. The delayed fracture regions consisted of intergranular facets, and the tendency of intergranular facture decreased in the order of 0.6C–22Mn, 0.6C–18Mn, and 0.6C–18Mn–2Al steels. Thus, the delayed fracture behavior was closely related with the intergranular fracture mode caused by grain boundary cementites. The addition of Al remarkably increased the resistance to delayed fracture because it suppressed the formation of grain boundary cementites and reduced the residual stresses in the cup specimen.

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#### 1. Introduction

Highly deformable steel sheets such as TWinning Induced Plasticity (TWIP) steels have been actively developed in automotive industries to reduce CO<sub>2</sub> and increase efficiency [1,2]. High-Mn TWIP steels are composed of single phase of austenite, which is stable at room temperature, as contents of austenite stabilizers such as Mn and C are quite high. Here in high-Mn TWIP steels, deformation twins formed during deformation prevent the movement of dislocations as they work for the grain boundary refinement, which is known as Hall-Petch effect [3–5], and the necking is suppressed during deformation due to the high work hardening rate. Thus, TWIP steels show high strength and ductility simultaneously [6].

Despite their superior tensile properties to other automotive steels, the commercialization of high-Mn TWIP steels is delayed because they are easily subjected to the cracking during forming or to the delayed fracture after forming [7–9]. However, the decrease of Mn content is not easy because of the stabilization of austenite at room temperature and the sufficient formation of twins. Researches on Al addition have also been conducted to prevent

delayed fracture and to improve formability. Al plays an important role in decreasing twin formation because it works for increasing stacking fault energy (SFE) [10–12]. These Al-added TWIP steels have excellent formability due to the decreased twin formation and the increased slip activation, which results in excellent combination of strength, ductility, and formability over TRansformation Induced Plasticity (TRIP) steels or dual phase steels [6,7,13].

Chin et al. [8] reported the cracking or delayed fracture took place in a high Mn TWIP steel during the cup forming test, whereas it did not occur in an Al-added TWIP steel, although the strength and elongation of the high Mn steel were higher than those of the Al-added steel. This was because the stress was concentrated on a certain location of the cup side of the high Mn steel. In the Al-added steel, the cracking or delayed fracture did not occur in the cup specimen as the twinning occurred more homogeneously. Berrahmoune et al. [14] found the delayed fracture after deep drawing of 301LN austenite steels, and explained it by the increased amount of martensite and the localization of residual stress. Recently, according to researches of Chun et al. [15] and Koyama et al. [16,17], the intergranular fracture occurred at hydrogen-introduced surface regions during slow strain rate tests of high-Mn TWIP steels. The introduction of hydrogen weakens grain boundaries in various types of steels, and thus induces the intergranular fracture [18-22]. However, the TWIP steels were

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often subjected to delayed fracture including intergranular fracture after they were exposed to the air for a long time without artificially introducing hydrogen [8,23]. This implies that the delayed fracture can be attributed to the intergranular embrittlement without artificially introducing hydrogen. In addition, TWIP steels in which the intergranular fracture is restricted show the better resistance to delayed fracture under hydrogen-introducing environments [23]. Thus, the delayed fracture of TWIP steels are closely related with the weakening of grain boundaries, and the resistance to intergranular fracture should be enhanced for preventing the delayed fracture, irrespective of effects of hydrogen introduction.

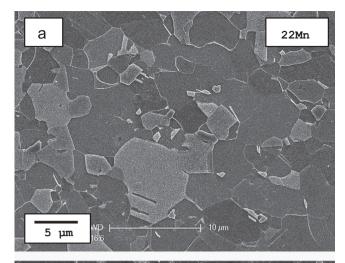
It is well known in conventional steels that the segregation of precipitates or inclusions on grain boundaries acts as an important reason for intergranular fracture [24]. Jin et al. [12] found the intergranular precipitation of fine M<sub>3</sub>C-type carbides by a nano secondary ion mass spectrometer (Nano-SIMS) analysis of TWIP steels, but its effects on mechanical properties or fracture behavior were not explained. However, exact mechanisms behind the delayed fracture are not sufficiently understood in relation to microstructures. This is because detailed analyses on amounts of twinning and slip [25,26], precipitation or segregation on grain boundaries [27,28], distribution of residual stresses varied with deformed locations [14,26], and hydrogen embrittlement are essentially needed [29,30]. In addition, alloying effects on microstructural modification processes during cup forming and delayed fracture tests are not analyzed in detail. From these understandings on effects of Al addition in TWIP steels, it is possible to prevent or minimize the delayed fracture of TWIP steels.

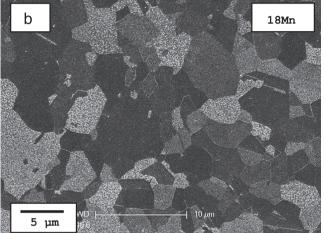
The delayed fracture behavior related with intergranular precipitation or segregation of two high-Mn TWIP steels and an Aladded TWIP steel was investigated in this study. The delayed cracking behavior was examined by dipping tests of the cup specimens in the boiled water, after which the microcrack formation behavior was analyzed. In addition to the experimental approach, equilibrium phases existing at high temperatures were verified by thermodynamic calculations, and the quantitative data related with carbide formation were compared with experimentally obtained data. Based on the results of microstructural analysis and intergranular embrittlement, effects of Al addition on improvement of delayed fracture properties were clarified.

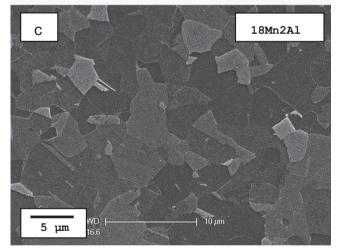
#### 2. Experimental procedure

Chemical compositions of the three TWIP steels are 0.6C-18Mn, 0.6C-22Mn, and 0.6C-18Mn-1.9Al, which are referred to as '18Mn', '22Mnl', and '18Mn2Al', respectively, for convenience. These steels were fabricated by a vacuum induction melting method. In the 18Mn2Al steel, 1.9 wt% of Al is added to increase the stacking fault energy (SFE) [10-12]. After thick plates of 30 mm in thickness were homogenized at  $1150~^{\circ}$ C for 1 h, they were hot-rolled at  $1100~^{\circ}$ C to produce 2.5-mm-thick sheets. The finish rolling temperature was  $900~^{\circ}$ C. The hot-rolled steel sheets were water-cooled to  $450~^{\circ}$ C, rolled at this temperature to make 1.4-mm-thick sheets, held at  $800~^{\circ}$ C for 30 s, and then water-cooled.

The steel sheets were polished and electro-etched in an etchant of 5% perchloric acid+95% acetic acid, and microstructures of longitudinal-transverse (L-T) plane were observed by a field emission scanning electron microscope (FE-SEM, model; XL30S FEG, Philips FEI, USA). Phases present in the steels were identified by energy dispersive spectroscopy (EDS). Nano-sized particles precipitated along grain boundaries were identified by transmission electron microscopy (TEM). For conventional TEM observation, specimens were mechanically polished to a thickness of  $100 \, \mu m$ , punched to prepare disc specimens (diameter; 3 mm)







**Fig. 1.** SEM micrographs of the (a) 22Mn, (b) 18Mn, and (c) 18Mn2Al steels. Electroetched in an etchant of 5% perchloric acid +95% acetic acid.

by a disc cutter, and then electro-etched in an etchant of 7% perchloric acid+93% acetic acid by a twin-jet polisher (model; TenuPol-5, Struers, USA) to prepare thin foil specimens. The thin foils were observed by a TEM (model; 2100, Jeol, Japan) operating at an acceleration voltage of 200 kV. In order to precisely analyze nano-sized grain boundary precipitates, thin foil specimens were made by a focused ion beam (FIB, model; Helior, FEI, USA), and were observed by a high-resolution TEM (model; JEM-2200FS (with Image Cs corrector), Jeol, Japan) operating at an acceleration

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