

Extended Abstract of PSA-19

## O-8\* SIMS investigation of internal hydrogen behavior of TWIP steel

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Austenitic high Mn twin-induced plasticity (TWIP) steels have been widely investigated due to its excellent combination of strength and ductility [1]. However, its high susceptibility of hydrogen embrittlement (HE) limits the wide spread of TWIP steels in producing structural components such as oil pipeline, LNG tank, and offshore structure. Therefore, many investigations have been made to elucidate the mechanisms responsible for the HE, and to alleviate HE either by alloy addition [2] or microstructure modification [3]. Nevertheless, there have been many issues to be clarified, such as (1) precise analysis on the amount of hydrogen, which is responsible for the HE, (2) interaction of hydrogen and various defects (dislocations and grain boundaries) and so on.

Visualization of local presence of hydrogen is a difficult task due to its low mass, very low detection sensitivity, very low concentration and high mobility [4]. Various approaches have been made for direct observation of hydrogen in metallic materials, hydrogen microprint technique [5], silver reduction and decoration [6], autoradiography [7], secondary ion mass spectrometry (SIMS) [8] and so on. However, limited work has been made on interactive relationship between spatially resolved hydrogen and associated specific microstructure defects such as dislocation. The experimental evidences behind this relationship enables us to understand the underlying mechanisms of HE because pronounced microstructure dependence of the damage mechanism is related to the degree of hydrogen trapping and localization [9].

Internal hydrogen distribution in TWIP steel has been spatially resolved on the basis of  $^1\text{H}$  ion depth profiling and direct  $^1\text{H}$  ion imaging principles using SIMS 7f Auto-Resistive anode encoder (RAE) and well formulated the hydrogen distribution by acquiring direct  $^1\text{H}$  ion images [3]. Spatially resolved hydrogen can be elucidated the hydrogen embrittlement behavior depending on the chemistry and microstructure of the TWIP steel. One of the remaining study on the H investigation by SIMS-RAE is the spatially resolved hydrogen content of the direct  $^1\text{H}$  ion images. Previous study of SIMS-RAE quantification have been successfully performed by current author for boron concentration in friction stir welding (FSW) steel [10]. Four standard reference materials (SRM) which is used for LECO RH-600 hydrogen analyzer, Fe-Cr-Ni 0.7H, 1.9H, 3.5H, 10.4H were prepared for SIMS and obtained the each relative sensitivity factors (RSFs) for H quantification using  $\text{Cs}^+$  primary ions and negative secondary at 15 keV, 100 nA and summarized in Table 1 where  $I_m$  is matrix ion ( $^{56}\text{Fe}^-$ ) intensity,  $I_i$  is impurity ion ( $^1\text{H}$ ) intensity, and  $\rho_H$  is hydrogen atom density in atoms/cm<sup>3</sup>.

From the result of linear fitting, mean RSF was determined to be  $7 \times 10^{18}$  atoms/cm<sup>3</sup> as shown in Figure 1. Unlike boron quantification by SIMS-RAE, monoatomic ions of  $^1\text{H}$  and  $^{56}\text{Fe}$  detection are more reliable than polyatomic ions of  $^1\text{H}^{16}\text{O}$  and  $^{56}\text{Fe}^{16}\text{O}$  detection using  $\text{Cs}^+$  primary ion bombardment as shown in Figure 2. It is suggested that the polyatomic ion species of  $\text{X}^{16}\text{O}$  can be utilized under  $\text{O}_2^+$  primary ion bombardment to reduce the matrix effect dramatically. The criterion for the selection of primary  $\text{Cs}^+$  or  $\text{O}_2^+$  is governed by ionization potential (IP) and electron affinity (EA) of the ion species, where hydrogen  $\text{EA}_{\text{H impurity}}$  is 0.75 and  $\text{IP}_{\text{Fe matrix}}/\text{IP}_{\text{H impurity}}$  is 0.58, so  $\text{Cs}^+$  for negative secondary is selected in this study.

Table 1. H concentration of SRMs and calculated RSFs.

\*  $I_m$ : matrix ion intensity,  $I_i$ : impurity ion intensity,  $\rho_H$ : H atom density in atoms/cm<sup>3</sup>

| SRM (ppm) | $I_m$   | $I_i$   | $\rho_H$ | RSF (atoms/cm <sup>3</sup> ) |
|-----------|---------|---------|----------|------------------------------|
| #1 (0.7)  | 16152.6 | 1008.9  | 3.27E18  | 5.24E19                      |
| #2 (1.9)  | 15919.3 | 2704.57 | 8.89E18  | 1.51E18                      |
| #3 (3.5)  | 15873.2 | 2984.4  | 1.64E19  | 3.08E18                      |
| #4 (10.4) | 15050.4 | 4446.8  | 4.87E19  | 1.44E19                      |

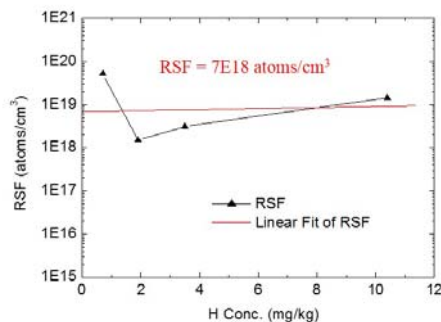


Fig. 1. Results of RSF for SRMs.

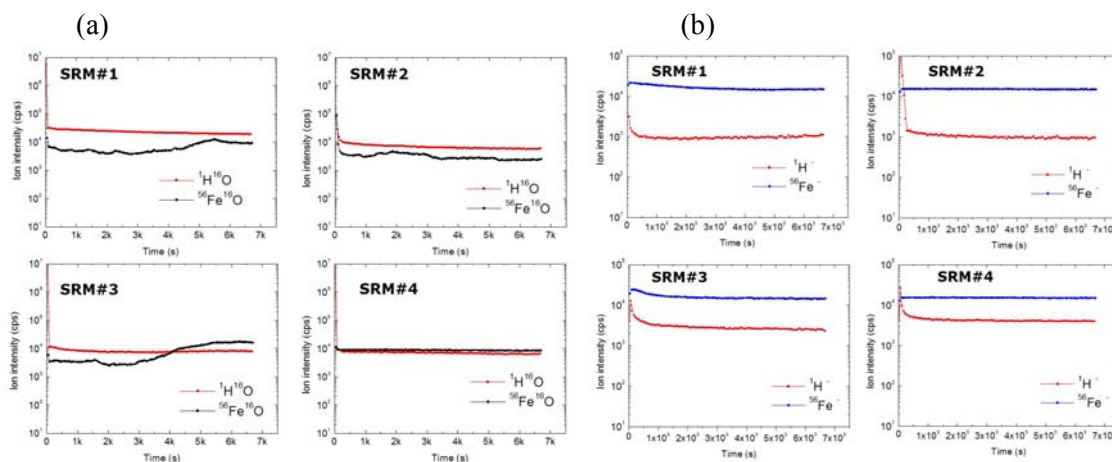


Fig. 2. Impurity ion (H) and matrix ion (Fe) intensity profile; (a) using polyatomic ion of  $^1\text{H}^{16}\text{O}^+$  and  $^{56}\text{Fe}^{16}\text{O}^+$ , (b) using monoatomic ion of  $^1\text{H}^+$  and  $^{56}\text{Fe}^+$ .

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