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Mechanical characterisation of microstructural evolution in 304 stainless steel subjected to high-pressure torsion with and without hydrogen pre-charging

Yoji Mine [a,*], Kaoru Koga [a], Kazuki Takashima [a], Zenji Horita [b,c]

[a] Department of Materials Science and Engineering, Kumamoto University Kurokami, Chuo-ku, Kumamoto 860-8555, Japan
[b] Department of Materials Science and Engineering, Kyushu University Motooka, Nishi-ku, Fukuoka 819-0395, Japan
[c] WPI, International Institute for Carbon-Neutral Energy Research (WPI-I2CNER), Kyushu University Motooka, Nishi-ku, Fukuoka 819-0395, Japan

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ABSTRACT

Micro-tensile tests were employed on a 304 metastable austenitic stainless steel to mechanically characterise the microstructures developed by processing through high-pressure torsion (HPT) with and without hydrogen pre-charging. The martensite formed by HPT processing of hydrogen-containing austenite exhibited low yield and tensile strengths but a high reduction of area compared to the one processed in the absence of hydrogen. This may be because dynamic martensite formed with hydrogen contains more retained austenite. Hydrogen charging into the austenite allowed the formation of ε–martensite, instead of deformation twinning, as an intermediate phase in the transformation to α′–martensite, which led to variation in the plastic behaviour. The inhomogeneity of the microstructure and the defects produced by deformation with hydrogen build a foundation but hardly play a crucial role in the hydrogen embrittlement (HE) of metastable austenitic steels. Excess hydrogen due to the dynamic martensitic transformation of hydrogen-containing austenite localises deformation in the retained austenite between the martensite regions formed, leading to the HE of metastable austenitic steels.

1. Introduction

With increasing requirements to reduce carbon dioxide emissions, the use of hydrogen as an energy carrier has recently become more practical. However, most metals used in harsh environments, e.g., in the marine, aerospace, nuclear, and chemical industries, often suffer from hydrogen embrittlement (HE). Unlike low-temperature embrittlement, which is accompanied by typical brittle fracture features, the fracture morphology after plastic deformation processes [1–4] has hindered the elucidation of the hydrogen-induced degradation of the mechanical properties. Therefore, studying hydrogen-induced plasticity is important to understand the HE mechanism. A previous study by Takai et al. [5] revealed that the defects produced by deformation with hydrogen, rather than the hydrogen itself, play an essential role in the hydrogen-induced degradation of the ductility in iron with a body-centred cubic (bcc) structure or in Inconel 625 with a face-centred cubic (fcc) structure. For austenitic stainless steels of interest, HE becomes more intense with a lower stability of the austenitic phase [6–9]. Moreover, the change in the crystal structure from fcc to bcc through deformation, i.e., the deformation-induced martensitic transformation, complicates the HE phenomenon in metastable austenitic steels. We focused on the mechanical characteristics of the microstructure developed by deformation in a hydrogen-containing metastable austenitic steel. A previous study by Mine et al. using high-pressure torsion (HPT) processing on hydrogen-pre-charged 304 and 316L stainless steels [10] revealed that solute hydrogen reduced the deformation-induced α′–martensitic transformation, which resulted in a reduction in the microhardness. The decreased microhardness in the hydrogen-pre-charged microstructure was attributed to its low fraction of the α′–martensitic phase. However, the solute hydrogen not only reduced the deformation-induced martensitic transformation but might also have affected the development of the resulting microstructure, as reported by Takai et al. for stable phase metals [5].

In the case where a second phase such as α′–martensite is dynamically formed within the parent phase, it is difficult to evaluate the mechanical characteristics of each phase using conventional mechanical testing. In contrast, micro-tensile testing enables the evaluation of the strength and ductility properties of microstructural constituents on the order of several tens of micrometres [11–16]. The objective of this study was to clarify how the defects produced through deformation with hydrogen contribute to the HE process of a metastable austenitic stainless steel.

* Corresponding author.
E-mail address: mine@msre.kumamoto-u.ac.jp (Y. Mine).

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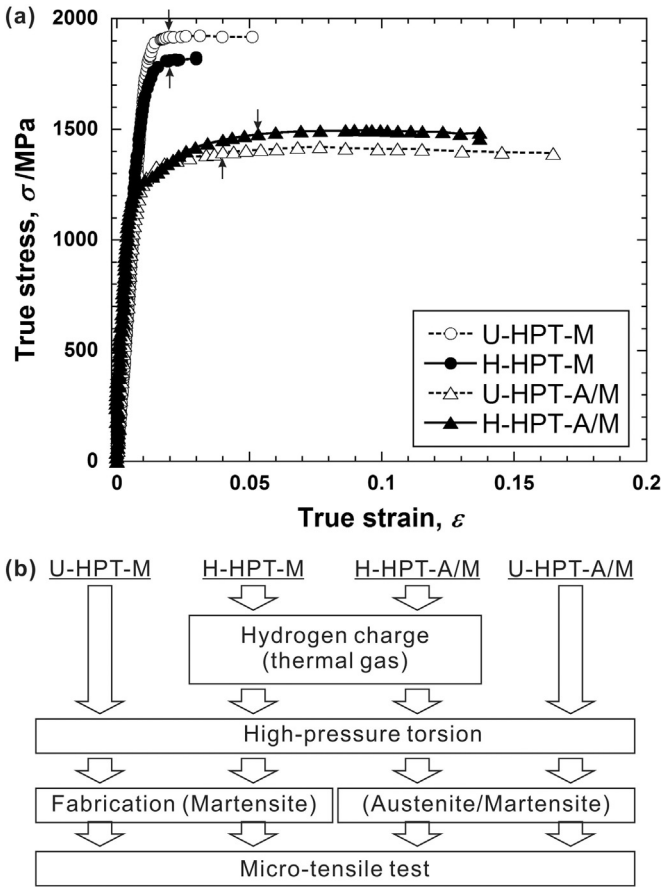

Fig. 1. (a) True stress–strain curves for the martensite and two-phase specimens and (b) their respective processing histories. The arrows in (a) indicate the onsets of necking.

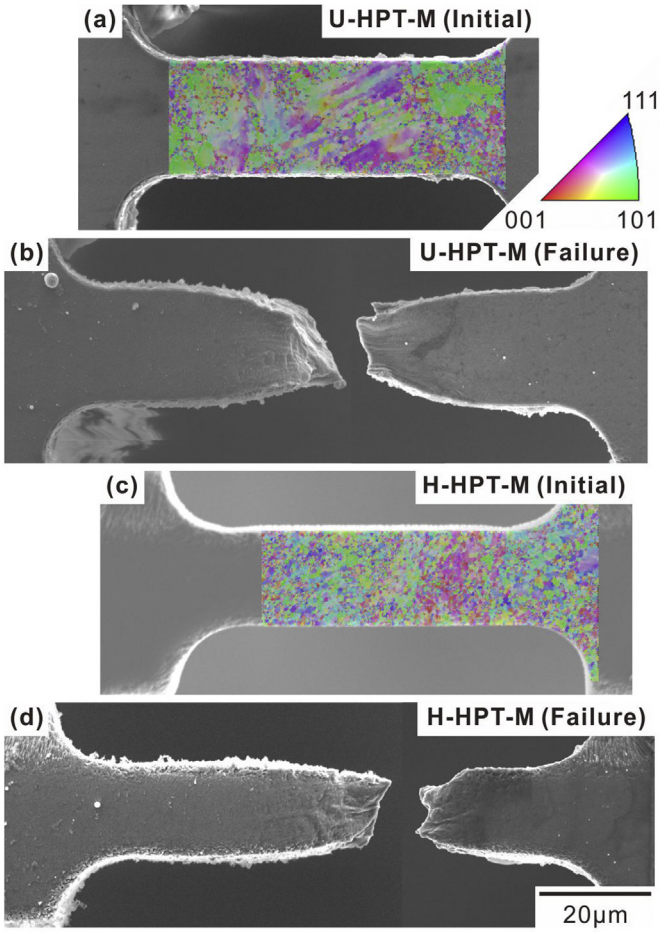

Fig. 2. (a, c) EBSD maps overlapped on the corresponding SEM images taken in the initial state and (b, d) fracture morphologies of the martensite specimens.

For this purpose, micro-tensile testing was employed for deformation-induced martensite specimens and retained austenite specimens prepared from the microstructures developed by HPT-processing of a type 304 austenitic stainless steel, with and without hydrogen pre-charging.

2. Material and experimental methods

The material used in this study was a 304 (JIS-SUS304) commercial austenitic stainless steel, which was composed of 0.05C, 18.54Cr, 8.09Ni, 0.58Si, 1.24Mn, 0.025P, and 0.003S (in mass%), with the remainder being Fe. It was received in the form of a plate that was 30 mm in thickness after solution treatment. The Vickers hardness in the as-received condition was 176 ± 10, where the error range represents the 95% confidence interval.

Disc-shaped samples with a diameter of 19 mm and an approximate thickness of 0.8 mm were machined from the plate. Discs with and without hydrogen pre-charging were processed by HPT at room temperature in air; the corresponding discs are denoted as H-HPT and U-HPT discs. Hydrogen pre-charging was undertaken at a temperature of 543 K by exposure to hydrogen gas for 200 h at a pressure of 10 MPa. These conditions for charging were sufficient to provide a nearly uniform hydrogen distribution throughout the thin specimen. The saturated hydrogen content was determined to be ~25 mass ppm. The HPT facility consisted of a pair of tool-steel anvils having a centric shallow circular cavity with a diameter of 20 mm and a depth of 0.25 mm. Shear strain was imposed by rotating the lower anvil with respect to the upper one for one turn at a rotation speed of 1 rpm under a pressure of 1.5 GPa.

Table 1
The fraction of α′–martensite, yield strength, tensile strength, and reduction of area for the martensite and two-phase specimens.

	α′–martensite fraction (%)	Yield strength (MPa)	Tensile strength (MPa)	Reduction of area (%)
U-HPT-M	> 99	1820	1880	79
H-HPT-M	> 99	1690	1780	87
U-HPT-A/M	27	1190	1340	79
H-HPT-A/M	59	1240	1400	86

The microstructural evolution due to HPT processing was examined by electron backscatter diffraction (EBSD) analysis. The surface of the HPT-processed discs was finished by electro-chemical polishing. The crystal orientation was determined by automatic beam scanning with a step size of 0.08–0.2 μm at an accelerating voltage of 20 kV in a field emission gun scanning electron microscope (SEM) using EBSD patterns and TSL orientation imaging microscopy software (OIM v. 7.1.0). The sample for transmission electron microscopy (TEM) was milled using a focused ion beam (FIB). The TEM observation was performed with a JEOL JEM-2000FX system operated at an accelerating voltage of 200 kV.

Gauge sections with dimensions of 50 μm × 20 μm × 20 μm of micro-tensile specimens were fabricated using FIB from the austenitic and α′–martensite phase regions and two-phase region in the H-HPT and U-HPT discs. The micro-tensile specimens of the