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## Criteria for determining hydrogen compatibility and the mechanisms for hydrogen-assisted, surface crack growth in austenitic stainless steels



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#### ABSTRACT

To establish novel criteria for determining the hydrogen compatibility of austenitic stainless steels, as well as to elucidate the mechanisms for hydrogen-assisted surface crack growth (HASCG), slow strain rate tensile (SSRT), elasto-plastic fracture toughness ( $J_{IC}$ ), fatigue crack growth and fatigue life tests were performed on Types 304, 316 and 316L steels in high-pressure hydrogen gas. As a criterion for the use of austenitic stainless steels with lower austenitic stability in hydrogen gas, a reduction in area (RA) in hydrogen gas,  $\varphi_{\rm H} \ge 57\%$ , or a relative reduction in area, RRA  $\ge 0.68$ , is proposed to ensure that there is no degradation in tensile strength by hydrogen. Observation of fracture surface morphologies and crack growth behaviours demonstrated that, in high-pressure hydrogen gas, SSRT surface crack grew via the same mechanism as for  $J_{IC}$  crack and fatigue crack, i.e., these cracks successively grew with a sharp shape under the loading process, due to a localized slip deformation near the crack tip. Based on the elucidated HASCG mechanism, total elongation in hydrogen gas,  $\delta_{\rm H} \ge 10\%$ , or,  $\varphi_{\rm H} \ge 10\%$ , is also introduced as another criterion. © 2015 Elsevier Ltd. All rights reserved.

#### 1. Introduction

Fuel cell vehicles (FCVs) have just been commercialized in Japan, and constructions of hydrogen fueling stations have also been promoted. For safety use of such systems, it is necessary to properly control the strength degradation of mechanical components used in hydrogen environment and to perform strength design of the components in consideration for the detrimental effect of hydrogen [1,2].

In the material selection for metallic components used in high-pressure hydrogen gas, relative reduction in area (RRA) has often been used as a criterion for characterizing the hydrogen embrittlement (HE) [3–10]. The RRA is obtained via a slow strain rate tensile (SSRT) test, which defines the ratio of a reduction in area (RA) in hydrogen gas,  $\omega_{\rm H}$ , to a RA in inert gas,  $\varphi_{\rm r}$ , i.e., RRA =  $\varphi_{\rm H}/\varphi_{\rm r}$ . Yamada and Kobayashi [8] proposed the following relational expression for qualifying 300-series austenitic stainless steels (e.g., Types 316 and 316L) for their use in hydrogen gas:

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Nomenclature	
а	crack length
Α	cross-sectional area after SSRT test
$A_0$	cross-sectional area before SSRT test
da/dN	FCG rate
$(da/dN)_{H}$	FCG rate in hydrogen gas
$(da/dN)_{H}$	/(da/dN) relative FCG rate
$\Delta K$	stress intensity factor range
$\delta_{\rm H}$	total elongation in hydrogen gas
Ε	Young's modulus
3	nominal strain of SSRT specimen
f	test frequency
$\varphi$	RA in inert gas
$\varphi_{\rm H}$	RA in hydrogen gas
$arphi_{H,cal}$	calculated RA in hydrogen gas with nominal strain
$\varphi_{\rm H,U}$	RA in hydrogen gas based on the cross-sectional area of the uniform deformation region
J	J-integral
Jic	elasto-plastic fracture toughness
l	gauge length after SSR1 test
	gauge length before SSR1 test
N <sub>f</sub>	
N <sub>p</sub>	rug life
NI <sub>eq</sub>	nickei equivalent
ĸ	stics fallo
3	applied stress
<i>0</i>	allowable design stress
σ <sub>allowable</sub>	tensile strength of a material
V V	cross-head speed
v	Poisson's ratio
Ŵ	specimen width of CT specimen

(RA obtained via tensile testing in inert gas or RA as reported in an inspection certificate) × RRA

 $\geq$  (Minimum requirement for RA specified in JIS standard)

In most of the materials used in the analysis by Yamada and Kobayashi, the RA values under air exceeded 75% [3]. On the other hand, according to Japanese Industrial Standards (JIS G 4303), the minimum requirement for RA is 60% for 300-series stainless steels [8]. Based on the two preceding facts, Yamada and Kobayashi required the 300-series stainless steels to satisfy RRA  $\ge 0.8$  (=60/75), i.e., minor hydrogen embrittlement was tolerated for the materials.

(1)

(3)

Moreover, using existing literature, Yamada and Kobayashi [8] analysed a series of RRA data for Types 304, 316 and 316L, showing that a relationship between the RRA and the nickel equivalent content ( $Ni_{eq}$ ) in 70-MPa hydrogen gas at -40 °C was successfully fitted by the following equation:

$$RRA[\%] = A + B \tanh\{(Ni_{eq} - D)/C\}$$
(2)

where *A* = 60, *B* = 40, *C* = 1.339 and *D* = 27.76. The Ni<sub>eq</sub> [mass%] is calculated by [11]:

$$Ni_{eq} = 12.6 C + 0.35 Si + 1.05 Mn + Ni + 0.65 Cr + 0.98 Mo$$

where the unit of the elements is mass%. From Eq. (2), it is calculated that RRA  $\ge 0.8$  is equivalent to Ni<sub>eq</sub>  $\ge 28.5$  mass%.

On the basis of the analyses described by Eqs. (1)–(3), Yamada and Kobayashi [8] suggested that 300-series austenitic stainless steels, satisfying both  $\varphi \ge 75\%$  in inert gas and Ni<sub>eq</sub>  $\ge 28.5$  mass%, are eligible for use in hydrogen gas up to 70 MPa, at temperatures ranging from -40 °C to 85 °C. For the infinite fatigue life design of components, it is noted that fatigue limit should not be degraded in hydrogen gas, in addition to the above requirement based on the RRA.

In order to improve the economic efficiency of the 70-MPa hydrogen station, lower-cost, austenitic stainless steels with less austenitic stability, as well as low-alloy and carbon steels, are greatly sought for use by industry. However, it has been reported that these steels do not satisfy RRA  $\ge 0.8$  [3,10]. Consequently, Matsunaga et al. [12] proposed the concept of no degradation in tensile strength (TS) by hydrogen as another criterion for determining the hydrogen compatibility of steels

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