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# Hydrogen susceptibility of nano-sized oxide dispersed austenitic steel for fusion reactor



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#### HIGHLIGHTS

- Hydrogen susceptibility of 316L ODS alloy was studied *in situ* under high-pressure gaseous hydrogen.
- Elastic modulus and yield strength were not affected by hydrogen exposure.
- Type-316L ODS-D150 showed the robust tensile properties under gaseous hydrogen.

#### GRAPHICAL ABSTRACT



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#### ABSTRACT

Effects of hydrogen on the mechanical properties and fracture patterns of nano-sized yttria ( $Y_2O_3$ ) dispersed 316L austenitic steel usually mentioned as oxide dispersed strengthened (ODS) steel was studied from *in situ* tensile test using small specimens under high-pressure hydrogen gas, and the steel's hydrogen desorption properties were quantified using thermal desorption spectra analysis. The steel samples were prepared using powder metallurgy from precursor powders with 5  $\mu$ m and 150  $\mu$ m particle size, then processed by mechanical alloying and hot isostatic press. The original powder size affected mechanical properties and hydrogen embrittlement. Type-316L ODS steel with fine grain size showed the robust tensile property under gaseous hydrogen and less hydrogen susceptibility. Yttria-doped 316L steel is a candidate for use as a structural component exposed to hydrogen in nuclear fusion reactors, so these results will be useful to material and component design criteria.

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#### 1. Introduction

Austenitic stainless steel has been widely applied to the various industrial uses due to its workability, weldability and corrosion resistance. An oxide dispersed strengthened (ODS) austenitic stainless steel has been developed for use as a structural material for gas turbines in the aerospace industry, and in fast breeder reactors in the nuclear power industry [1-8]. The structural materials for next-generation fission systems (Generation IV) and fusion reactor systems (DEMO) require superior mechanical properties (especially, reduced creep under high temperature) and resistance to very high temperature and radiation. For these reasons, high-strength ODS steel is a promising material candidate for the blanket structure of fusion reactor systems exposed to high temperature and intense neutron radiation [9]. A fusion reactor generates highly-concentrated hydrogen, which can attack materials and cause corrosion and fracture; these phenomena can occur in the blanket structure and in any parts and components that are exposed to hydrogen in fusion reactor system. Especially, hydrogen production by the (n, p) transmutation reaction (predicted hydrogen-production rate: 0.9 wppm/dpa under 14-MeV neutron irradiation) in first wall-structure is a contributor to hydrogen corrosion [10,11] in fusion reactors.

Type-316L austenitic stainless steel (STS) will be applied to various parts and components (e.g., valves, pipes, vessels, tubes, fittings, heat exchanger) of fusion reactors because it has sufficient tolerance for operation temperature and radioactivity intensity. Hydrogen effects and hydrogen-induced phenomena such as hydrogen embrittlement (HE) of Type-316L ODS steel with the resistance of high does irradiation and the high temperature should be investigated to determine whether this steel can ensure reliable operation of advanced reactor systems. Most of the research about hydrogen effects on ODS materials has been conducted using electrochemical hydrogen charging [9,10,12]; this method has the limitation that the hydrogen accumulates only near the material surface.

This study uses the high-pressure gas-charging method to quantify the effect of HE on nano-sized yttria  $(Y_2O_3)$  dispersed 316L austenitic steel. *In situ* tensile test using small specimen test technique were conducted in a hydrogen environment to investigate the mechanical properties. Fractography analysis was conducted, and thermal desorption behavior of hydrogen was studied.

#### 2. Experimental

Two raw type-316L austenitic steel gas-atomized powders (reported mean particle sizes 5  $\mu$ m (D5) and 150 $\pm$ 45  $\mu$ m (D150), Table 1; SANDVIK) and nano-sized yttria (Y<sub>2</sub>O<sub>3</sub>, Aldrich; reported mean particle size <50 nm) were used.

Mechanical alloying (MA) was used to obtain type-316L ODS steel powder. Prepared metal powder (99.7 wt.%) and yttria (0.3 wt.%) were milled together in an environmentally-controlled Ar-filled container at 230 rpm for 10 h (Pulverisette 5; Fritsch). Powder and container treatment was also conducted in environment condition-controlled glove box. The prepared raw type-316L ODS steel powder was subjected to canning process, then degassed at 500 °C. A hot isostatic process (HIP) was conducted to produce a fully-densified type-316L ODS block (Fig. 1, left) at 1250 °C and 100 MPa. Two blocks were prepared, one (type-316L ODS-D5) from D5, and one (type-316L ODS-D150) from D150. Specimens for tensile test were prepared for comparative study.

A small tensile specimen was produced with gauge length 19.00 mm, diameter 4.00 mm  $\pm$  0.01 mm, and grip length 12.7 mm (Fig. 1, right); these dimensions were based on ASTM E8/E8 M. The specimen's elastic modulus, yield strength, tensile strength, reduction of area (RA) and elongation (EL) were tested under gaseous hydrogen at 10 MPa pressure in an environment materials testing system (KRISS). Testing conditions were: grip separation 21 mm, initial testing speed 0.0012 mm·s<sup>-1</sup>, nominal gage length 12 mm, strain gauge removal point 9%, strain endpoint 9%; these values were based on ASME G142 criteria.

To analyze the characteristics of hydrogen evolution, thermal desorption spectroscopy (TDS) with gas chromatography (GC, Agilent Technologies 7890A) system was conducted at a heating rate of 100 °C h<sup>-1</sup> to 900 °C. A flat TDS specimen (50 mm long, 10 mm wide, 1 mm thick) was placed in a TDS tube furnace, and the gas was analyzed at 3-min intervals using He as a carrier gas. A standard gas mixture of H<sub>2</sub> (5.64  $\mu$ mol mol<sup>-1</sup>) and He (balance) supplied by KRISS was used for calibration. TDS samples were charged for 5 d at 150 °C in 10-MPa hydrogen gas after preparing bare samples as

#### Table 1

Compositions	of the gas-ato	mized type-316L a	ustenitic steel powder

Mean particle size [µm]	Code	Elements	Elements [%]										
		Cr	Ni	Мо	Mn	Si	С	Р	S	Fe			
5	D5	16.1	10.7	2.1	1.25	0.77	0.03	0.01	0.01	Bal.			
150	D150	16.8	10.4	2.4	1.24	0.73	0.02	0.01	0.01	Bal.			



Fig. 1. HIP-processed type-316L ODS steel (left) and small tensile specimen for in situ gaseous hydrogen embrittlement test.

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