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Hydrogen charging process instrument

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HIGHLIGHTS

- The developed instrument is effective tool for H-charging of metallic materials.
- The H-charging process can be optimized depending on the specimen material.
- *In-situ* hydrogen charging during tensile testing can be performed.

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ABSTRACT

Economically attractive and safe process instrument for hydrogen charging from glow discharge plasma was developed. The unique shape of the ionizer allows to restrict the plasma dispersion and focus it on the sample. The instrument can be combined with a loading device for *in-situ* hydrogen charging during tensile testing and heating to an elevated temperature.

Successful results of hydrogen charging were obtained for ODS-EUROFER steel, which is one of the most promising structural materials for fusion reactors and Gen-IV nuclear reactors. The suitable parameters of hydrogen charging were found experimentally. The plasma exposure time for homogenization of hydrogen concentration in the certain shape of the specimen of the ODS-EUROFER steel was determined to be more than 30 min. Comparative study of the surface morphology of the specimens before and after hydrogen plasma charging was performed by means of scanning electron microscopy (SEM).

1. Introduction

The components of the active zone of nuclear reactors are exposed to embrittlement effect by hydrogen or/and helium accumulated into the materials due to the (n, α) and (n, p) transmutation reactions. Thus, the structural materials of both fusion and fission nuclear reactors must be stable not only for the high temperature application, but also in the presence of hydrogen. The example of such a promising material is reduced activation ferrite–martensite (RAFM) ODS-EUROFER steel oxide-dispersion strengthened with 0.3 wt.% of yttrium oxide (Y_2O_3) nano-sized particles. The obtained microstructural properties of ODS-EUROFER steel satisfy the requirements of Gen-IV nuclear reactors in the resistance to high temperature creep, radiation-induced degradation and induced radioactivity. Also it is one of the promising materials for the application in ITER and DEMO fusion reactors, but its sensitivity to either hydrogen or helium embrittlement is still under research.

There are a number of methods of hydrogen charging such as high energy hydrogen ion irradiation, electrochemical hydrogen charging and hydrogen isotopes implantation from high voltage glow discharge or plasma exposure which are used for investigation of hydrogen uptake and trapping of different materials by means of thermal desorption spectroscopy (TDS) and transmission or scanning electron microscopy [1–4]. Based on these methods a new technical solution of hydrogen charging process instrument was developed and successfully tested for ODS-EUROFER and EUROFER 97 steels [5]. Furthermore, the developed technique can be easily combined with tensile loading and heating element, that allows to perform the tensile testing with continuous hydrogen charging at different temperatures.

2. Experimental

2.1. Preparation of the samples

The ODS-EUROFER steel was produced and provided for current research by Forschungszentrum Karlsruhe Institute for Materials Research [6]. Chemical composition of the studied steel is shown in Table 1.

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Table 1
Chemical composition of ODS-EUROFER steel, wt.%.

	C	Si	Mn	Cr	Ni	Mo	Al	W	V	Ti	Co
ODS-EUROFER	0.086	0.03	0.39	9.2	0.02	0.0056	0.003	1.14	0.1965	<0.003	0.0036

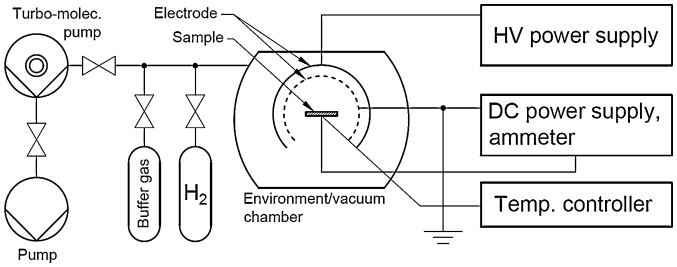

Fig. 1. Schematic diagram of the hydrogen charging process instrument.

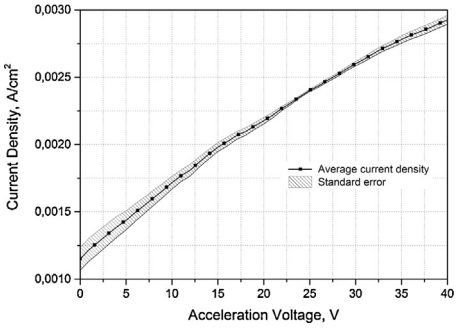

Fig. 3. Current density dependence on acceleration voltage.

Samples of ODS-EUROFER steel were cut by a spark-erosion machine from the hot-rolled plate. Hydrogen charged area of the specimens has a typical size of 6 mm × 4 mm × 0.4 mm. All the specimens were mechanically polished finishing with 3 μm diamond paste.

2.2. Hydrogen charging experiment

Schematic diagram of the hydrogen charging process instrument is shown in Fig. 1. The initial pressure of the environment/vacuum chamber is about 10^{-4} Pa. Charging pressure was experimentally found to be about 9×10^2 Pa of pure hydrogen atmosphere. The charging pressure can be slightly decreased by using the gas mixture of H_2 with an inert gas (buffer gas) such as Ar or He. Three electrode design of the H-charging instrument shown in Fig. 1 is used to control the hydrogen charging process. High voltage (HV) power supply generates the pulse output signal with amplitude of about 2 kV and frequency of about 15 kHz that produces the glow discharge between the outer electrode (anode) and inner electrode (cathode). Both electrodes are made of AISI 316 steel plain weave mesh with open area of 37%. Direct current (DC) power supply is used to accelerate the hydrogen ions, which pass through the reticulated inner electrode. The accelerating voltage between the inner electrode and the specimen can be varied, and the ion current from the specimen to the earth is measured. The temperature of the sample is measured using thermocouple imbedded into the holder of the specimen.

In order to restrict the plasma dispersion and focus it on the sample a unique shape of ionizer body was developed. Fig. 2a shows the shape and dimensions of the ionizer, which determine the distance between the outer and inner electrodes of about 4 mm, and between the inner electrode and the specimen of about 10 mm. The ionizer body should be non-conducting and thermally stable, and a ceramic material satisfies sufficiently these requirements. The elliptical shape of the ionizer promotes the homogeneous ion flow on the surface of the specimen. Specimen must be fixed along the dashed line shown in Fig. 2a. The internal structure of ionizer is shown in detail in Fig. 2b.

Ion current was measured for different applied acceleration voltages in the range from 0 to 40 V. The curve of calculated current density versus acceleration voltage in the given range is shown in Fig. 3. The ion current was found to be sufficiently stable in the range of accelerated voltage from 20 to 30 V, while the standard error of ion current measurement increases uniformly with either increasing or decreasing of the acceleration voltage from the stable range. In the whole tested range of the accelerated voltage the maximum deviation of the ion current was measured for the applied voltage close to zero. One can assume the deviations of ion current at lower and higher limits of the applied voltage associated with elastic collisions of ions and specimen surface and their reflection from the surface and/or sputtering of the specimen atoms, while in the range from 20 to 30 V the deviation of ion current is effectively suppressed.

Fig. 4 shows that the temperature of the specimen changes with the increase of the applied acceleration voltage as an exponential function. Increase of the applied voltage from 0 to 40 V results in temperature increase of about 10 °C, that is quite small to affect a significant increase of the hydrogen diffusivity. Also, the heat can be transmitted by external cooling of the specimen holder.

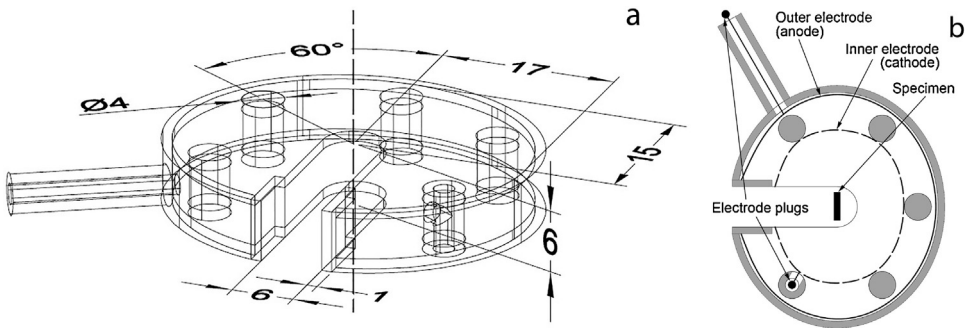

Fig. 2. Ionizer body dimensions (a) and its schematic construction (b).