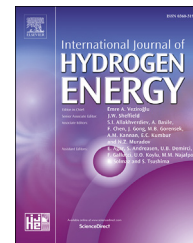




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Corrosion characteristics of a nickel-base alloy C-276 in harsh environments

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ABSTRACT

The oxidation behaviors of alloy C-276 in two harsh environments: high-temperature air and supercritical water (SCW), respectively representing the working conditions of the external and internal surfaces of reactors for SCW gasification biomass to produce H₂, were investigated. In two environments, all oxidation kinetics followed parabolic laws, while the corrosion rate of alloy C-276 exposed to supercritical water gasification (SCWG) environments was 2.5–3 times higher than that in high-temperature air. The oxide scale formed in air at 500 °C consisted of an outer Fe-rich layer (Fe₂O₃ and NiCr₂O₄) and an inner layer of Cr₂O₃ and NiCr₂O₄, while the outer Fe-rich layer disappeared as the temperature increased to 550 °C. Compared to the scales formed on nickel-base alloys in near-pure SCW, the absence of NiO and Ni(OH)₂ phases within the scales formed on the C-276 samples in present SCWG environment may be due to higher molar proportion of hydrogen.

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Introduction

Hydrogen has been considered one of the most potential clean energy alternatives to fossil fuels due to the dual concerns of energy security and global warming [1]. Hydrogen can be produced directly by gasification from renewable energy sources such as bio-solids, animal waste, agricultural residues, municipal wastes and sludge. Renewable energy sources gasification in supercritical water (i.e., supercritical water gasification, SCWG) is a promising technology producing hydrogen from high moisture biomass, which has many advantages over conventional biomass gasification processes,

such as high gasification efficiency and high molar fraction of hydrogen (35–45%) [1–4]. The most noteworthy is that, there is no need of a drying process before SCWG treatment for wet biomass even with a moisture content up to 95% [1].

The gasification of wet biomass in supercritical water (SCW) is a high-temperature (>400 °C) and high-pressure (>24 MPa) endothermic reaction. The SCWG reactor generally operates at 400–600 °C with an addition of catalysts or not [4], while for indirectly heated SCWG systems the temperature of the reactor' exterior surface contacting with near-still air can reaches 450–650 °C sometimes. Therefore, the damage issues of SCWG reactors are not only the corrosion of their inner wall in SCW environments, but also the high-

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temperature oxidation of the exterior surface exposed to high-temperature air. The corrosion behavior of the potential construction materials of SCWG plants in these two above-mentioned harsh environments needs to be understood. Compared with other alloys, the preponderance of nickel-base alloys in SCWG environments is evident, due to their generally superior corrosion resistance and strength at elevated temperatures, and prior experience with pure-SCW and supercritical water oxidation (SCWO) environments [4–9]. In near-pure SCW, extensive studies have been performed that describe the oxide scales formed on the nickel-base alloys, consisted of NiO/Ni(OH)₂ particles on a uniform, protective oxide layer composed of very fine Cr-rich oxides [8,10–12]. While the sample surfaces were generally covered by one single-layer NiO or a duplex-layer oxide scale (an outer predominant NiO layer and an inner Cr-rich oxides layer), when the nickel-base alloys were exposed to SCWO environments for a longer exposure time [5,7,13]. In contrast to SCWO and pure-SCW, the corrosion behavior of various nickel-base alloys exposed to SCWG environments has not been established in detail. Several references suggested or indicated that higher molybdenum content increases corrosion resistance of nickel-base alloys in SCWG environments. In addition, for the high-temperature oxidation of nickel-base alloys a majority of the available database has been generated in laboratory tests that was conducted at temperatures of >800 °C, higher than those at which the tested alloys would normally be used [14–17]. These experimental studies are seldom conducted at the practical service temperatures such as 450–550 °C, but which can provide useful information to instruct the engineering application of the potential materials.

Hastelloy C-276 is a nickel-base alloy with high concentrations of Cr and Mo, which has been successfully applied in the chemical engineering field due to its satisfactory corrosion resistance to HCl and in reducing conditions. The present work was intended to evaluate the corrosion behaviors of alloy C-276 as the candidate material for SCWG reactors in two harsh environments: high-temperature air and SCWG environments, which represent the working conditions of the external and internal wall of the SCWG reactor, respectively.

Experimental procedures

A commercial nickel-base alloy Hastelloy C-276 was analyzed in this work, of which the nominal chemical composition in weight percentage is given in Table 1. Rectangular samples with dimensions of approximate 14 × 14 × 6 mm were cut from the as-received cast C-276 bar. Before tests, the samples were progressively ground to 2000 grit with fine SiC emery papers, then polished using soft cloths impregnated with diamond paste of ~1 μm, after which they were ultrasonically washed in distilled water and degreased with acetone. In order to model the supercritical water gasification condition,

the practical wastewater used in SCW exposure tests contained chemical oxygen demand (COD) of 52000 ppm for the presence of organic compounds. Air and SCW exposure tests were performed in a conventional tubular furnace and one SCW experimental system, respectively. For high-temperature air experiments, the samples were placed inside the furnace which was set to 450, 500, and 550 °C for 10, 30, 60, 240, 500, and 1000 h, respectively. The detailed construction and system capabilities of the SCW apparatus are described in our previous paper [5,18,19]. Firstly, the SCW autoclave was loaded with the alloy samples and predetermined wastewater volume. Then, it was consecutively sealed, purged with nitrogen, and heated to experimental condition of 500 °C and 25 MPa. One tests finished until the corresponding exposure time of 10, 30, 60, 90, or 120 h was reached.

The corrosion characteristics of the samples were analyzed by means of gravimetry, scanning electron microscopy (SEM) equipped with an energy dispersive X-ray spectroscopy (EDS), Raman spectroscopy, X-ray Diffractometer (XRD), and X-Ray Photoelectron Spectrometer (XPS). An electronic balance (Satorius) with a measurable sensitivity of 0.1 mg was used for gravimetry. Two to three samples were used for each weight gain data, and the weight gain was normalized by the surface area of the samples (weight gain per unit area). The exposed sample surfaces were analyzed by A JSM-6390 SEM and EDS analyses when appropriate. A Raman spectroscopy (LabRAM HR 800) and an X'PertProMPD Diffractometer with Cu radiation were employed to determine the product phases. The X-Ray Photoelectron Spectrometer (Kratos AXIS-ULTRA DLD) was used to detect the composition vs. depth profile of the C-276 samples exposed to supercritical water gasification environment for 120 h.

Results and discussion

Corrosion in natural air

The weight gain (ΔW) of the alloy C-276 samples exposed to near-still air at 450 °C, 500 °C, and 550 °C for several exposure times were measured through gravimetry, as shown in Fig. 1. The weight gain increased with an increase in exposure time and temperature. At all temperature studied, after an initial period of almost linear rapid oxidation, a stage of steady-state oxidation occurred, indicative of the formation of a protective oxide scale. The oxidation kinetic of the samples is assumed to follow parabolic relationships that can be described by the equation:

$$\Delta W = (k_p t)^{1/2} \quad (1)$$

where k_p is the temperature-dependent oxidation rate in $\text{mg}^2 \text{cm}^{-4} \text{h}^{-1}$, ΔW represents the weight gain in mg cm^{-2} , and

Table 1 – Chemical composition of Hastelloy C-276 (in wt.%).

Alloy	Ni	C	Si	Cr	Mo	Fe	Co	W	V	P	S
C-276	Bal.	0.007	0.06	15.8	16.2	6.5	1.9	4.2	0.30	0.035	0.025

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