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Oxidation behavior of stainless steels 304 and 316 under the Venus atmospheric surface conditions

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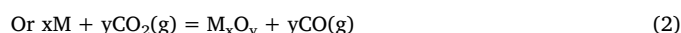
ABSTRACT

In general support of planetary exploration missions, the chemical, structural and microstructural behavior of stainless steels 304 and 316 was probed after exposure to a gas mixture with temperature, pressure, and composition mimicking the Venus lower atmosphere. Exposures were carried out in the Glenn Extreme Environments Rig (GEER) chamber with the Venusian gas mixture (96.5% CO₂, 3.5% N₂, 30 ppm H₂O, 150 ppm SO₂, 28 ppm CO, 15 ppm OCS, 3 ppm H₂S, 0.5 ppm HCl and 5 ppb HF) at 9.2×10^6 Pa and 740 K for a duration of 10, 21, and 42 days. Stainless steel 304 and 316 samples were characterized before and after the experiment by gravimetric analysis, X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), and cross-section electron microscopy analysis. All samples exposed to the Venusian atmosphere formed double-layered scales in which the outer layer is mainly magnetite (Fe₃O₄) and the inner layer is mainly spinel (Fe_{3-x}Cr_xO₄). Nickel sulfide and oxide formed at the interface of stainless steel 304. Nickel sulfide also formed in the inner and outer layer of the stainless steel 316 scale. Chromium carbide was only detected by Auger analysis at the interface of the unpolished side of the stainless steel 304 coupon sample. Stainless steel 304 with its smoother surface (780.62 nm total roughness on the polished side and 3.57 μm on the unpolished side) and slightly higher chromium and slightly lower nickel content, exhibited slower kinetics of oxidation ($3.1 \pm 0.5 \times 10^{-3}$ mg cm⁻² day⁻¹) than stainless steel 316 (0.05 ± 0.07 mg cm⁻² day⁻¹) with its rougher surface (4.78 μm total roughness on both sides). The slightly higher molybdenum content in stainless steel 316 was also responsible for increasing its kinetics of oxidation because of the formation of molybdenum sulfide. Our thermodynamic calculations of the phase assemblage containing the Venus gas mixture and the stainless steel samples predicted hematite Cr₂O₃(s), Fe₂O₃(s), Cr(s), Cr₂S₃(l), Ni(s), Fe(s), CrO(l), H₂(g), HCl(g), HF(g), NH₃(g) and H₂S(g) as the thermodynamically stable phases formed at the Venus atmospheric conditions.

1. Introduction

Degradation of materials by supercritical (SC) fluids in extreme environments is a current and future concern for understanding the stability of materials. This is important for a variety of applications, including the potential use of SC CO₂ as a coolant in nuclear reactors [1] and CO₂ in pipelines for possible capture and sequestration applications [2]. NASA's interest in SC CO₂ stems from various planetary science situations where SC CO₂ is encountered. This includes the surface of Venus, where the atmosphere is thought to be a primarily a gas-like (due to its high temperatures) form of SC CO₂ with smaller amounts of other gases. It is essential to understand the interactions of structural alloys with this atmosphere for both Venus simulation chamber construction and for future Venus lander construction [3,4].

The effects of pure SC CO₂ on alloys have been explored since the 1970s, due the use of SC CO₂ as coolant in nuclear power plants. This involves temperatures up to 873 K and pressures up to 2×10^7 Pa, which are somewhat higher than Venusian conditions. Nonetheless, these studies provide helpful background information. Similar to other high-temperature corrosion processes [5], corrosion in SC fluids involves transportation of CO₂ to the fluid/metal interface, reaction and formation of products at this interface, and then transportation of any additional products away from the surface [6–12]. This is shown schematically in Fig. 1. The chemical reactions for the mechanism in Fig. 1 can be written as follows:



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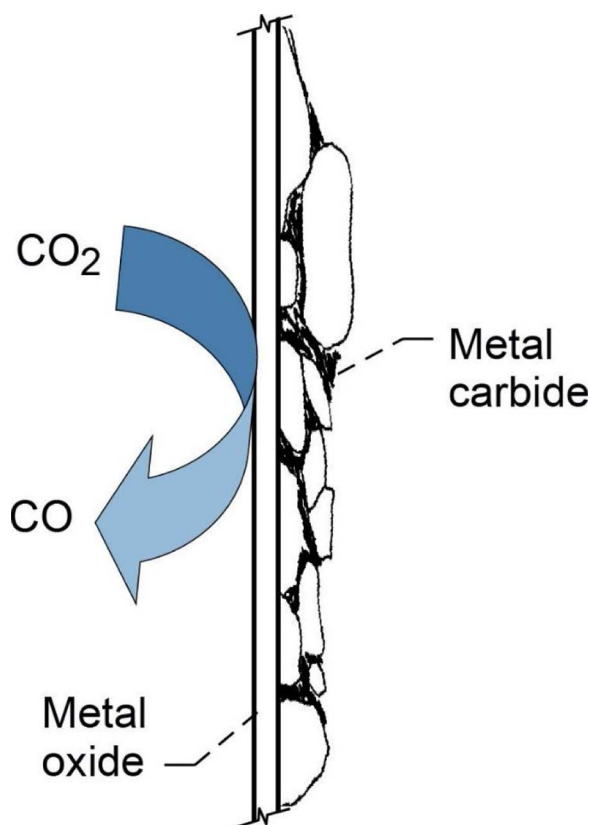


Fig. 1. SC CO₂ corrosion mechanism schematic.



M = Fe, Cr, and C^s = carbon in solution

Like high-temperature oxidation, a dense protective oxide (M_xO_y) is desirable. This would exhibit parabolic kinetics. One concern, however, is the dissolution of carbon in the underlying alloy at the grain boundaries, which may lead to a loss of strength.

Furukawa et al. [6] examined a 12Cr martensitic steel and 316 stainless steel in SC CO₂ from 673 to 873 K at 2×10^7 Pa for periods up to 2000 h. The 12Cr martensitic steel showed good parabolic behavior and an outer layer of Fe₃O₄ and inner layer of Fe(Fe_{(1-x)Cr_x)₂O₄. They also observed carburization of the base metal.}

Rouillard et al. [7,8] studied a 9Cr ferritic-martensitic steel and three austenitic steels (UNS S31603 (17Cr-11Ni steel), UNS S30815 (21Cr-11Ni steel) and UNS N08800 (20Cr-30Ni steel)) in SC CO₂ at 823 K and 2.04×10^7 Pa for 310 h. The 9Cr ferritic-martensitic steel showed a thick Fe-Cr duplex oxide scale composed of an outer magnetite layer, Fe₃O₄, and an inner spinel oxide, Fe(Fe_{(1-x)Cr_x)₂O₄, enriched with carbon. The austenitic steels showed a much thinner scale mainly composed of a single chromia, Cr₂O₃, or a mixed iron-chromium oxide.}

Pint et al. [9] performed some initial corrosion studies of a wide variety of Fe- and Ni-based alloys exposed to SC CO₂ at 673–1023 K and $1.2\text{--}3 \times 10^7$ Pa for 500 h. They found that iron rich oxides formed on 2–13 wt% Cr steels exposed to SC CO₂ above 400 °C and that thin protective chromium-containing layers formed on the austenitic steels.

Anderson, Sridharan and coworkers [10–12] obtained kinetics for a number of alloys in SC CO₂ at 823 K and 2.5×10^7 Pa for periods to 3000 h. They observe the best behavior with an oxide-dispersion strengthened (ODS) ferritic alloy (PM2000), followed by nickel-based alloys, and 201 and 316 stainless steels. Most of these appear to follow near parabolic kinetics with protective oxide formation. However, the data for 316 stainless steel show a lot of scatter, similar to the observations of Furukawa et al. [6]. They also see carburization in the

alloys, which appears to lead to scale spallation. Lim et al. [13] studied a range of steels in SC CO₂ at 923 K and $115\text{--}2.25 \times 10^7$ Pa for periods up to 3000 h. Alloys with a mixed alumina-chromia protective oxide showed the best behavior. Alloy 316 showed the least corrosion resistance.

Board and Winterborne [14] studied the long-term corrosion of fin-rolled AISI type 316 and 347 stainless steel tubing up to 5000 h of exposure at 923 K and 973 K and at pressures of 2.8×10^6 Pa and 4.1×10^6 Pa in CO₂ containing 1 vol.% CO and 200 ppm water vapor. Prior to the experiments, the samples were treated under different conditions (annealed in air and nitrogen at 1123–1353 K and abrasive cleaning). They found no appreciable weight gain differences between the samples exposed to different temperatures (923 K and 973 K), pressures (2.8×10^6 Pa and 4.1×10^6 Pa), and in dry or wet atmospheres. The weight gain of the samples is initially parabolic followed by linear (paralinear) kinetics. Carbide was detected at the grain boundaries of the air-annealed Type 316 steel after oxidation for 3000 h in CO₂ at 923 K. A Cr₂O₃ protective film formed on the cold-worked samples in CO₂ at 923 K and 973 K while low-temperature heat-treatment in nitrogen prior to oxidation resulted in a less protective spinel type oxide. Stainless steel 316 showed better corrosion resistance than stainless steel 347.

The studies on SC CO₂ corrosion of alloys for pipes in CO₂ capture and sequestration schemes are at lower temperatures (e.g., 323 K) and include various other gases such as H₂O, O₂, and SO₂ impurities [2]. At these temperatures, the SC CO₂ is more fluid-like as compared to higher temperatures where the SC CO₂ would be more gas-like. Nonetheless, the results are relevant to develop an overall understanding of the process. In general H₂O tends to promote corrosion. SO₂ and H₂O tend to increase corrosion rates due to formation of sulfurous acid.

The goal of this work is to study the chemistry, structure, microstructural, and kinetic properties of stainless steels 304 and 316 exposed to SC fluid consisting of chemical compositions and temperature and pressure conditions of the Venus atmosphere (96.5% CO₂, 3.5% N₂, 30 ppm H₂O, 150 ppm SO₂, 28 ppm CO, 15 ppm OCS, 3 ppm H₂S, 0.5 ppm HCl, and 5 ppb HF at 740 K and 9.2×10^6 Pa). In particular, these alloys were selected for this study because the GEER vessel is composed of stainless steel 304 and other small items (isolation valve, inner rings, line from the GEER vessel to isolation valve, etc.) inside of the pressure boundary are composed of stainless steel 316. Moreover, the knowledge gained in this study can be useful in similar scenarios involving these materials and carbon dioxide at high pressures and temperatures such as SC CO₂ as a coolant in nuclear reactors [1] and CO₂ in pipelines for possible capture and sequestration applications [2]. In addition, we use thermochemical modeling software to predict the corrosion products in this complex environment. The temperatures and pressures used in this study are somewhat less than those encountered in nuclear applications, and the addition of gases beyond CO₂ produces some important effects.

2. Materials and methods

2.1. Materials

The stainless steel 304 and 316 coupon samples of this work were provided by ATI Allegheny Ludlum (Midland, PA). The coupon samples dimensions were 5.2 by 4.1 by 0.2 cm with total surface area of 45 cm². The stainless steel 304 coupons were plates from hot-rolled coils annealed rework and the stainless steel 316 coupons were from cold rolled coils annealed during their manufacturing process. According to the manufacturer, one of the sides of the stainless steel coupon samples has a #8 mirror finish, which was achieved by polishing with at least a 320 grit belt or wheel finish. The chemical composition of the stainless steel samples is provided in Table 1. All samples were cleaned with a detergent solution, rinsed with deionized water, and ultrasonicated in acetone and ethanol prior to the experiments.

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