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Compact Reverse Water-Gas-Shift Reactor for Extraterrestrial In Situ Resource Utilization

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A compact reverse water-gas-shift reactor suitable for extraterrestrial use as part of the in situ propellant production system is reported. The reactor is less than 15 cm^3 in volume and weighs less than 50 g. With an Ru/ZrO_2-CeO catalyst it produces over 150 g H_2O/h operating at 800°C. This is near equilibrium conversion at about half-scale of a Mars sample-return mission. Even at these high processing rates, the pressure drop remains low (from 1.6 to 7.6 kPa).

Nomenclature

ΔH = heat of reaction

I. Introduction

THE next phase of manned space exploration may involve extended operation on extraterrestrial bodies, first the moon and eventually Mars and beyond. Use of indigenous resources, commonly referred to as in situ resource utilization (ISRU), is essential to the success of these missions. Other than solar power, no space missions have used available indigenous resources. Numerous studies have supported the use of in situ resources to reduce mission mass, cost, and risk for both robotic and human exploration, as well as to enhance, extend, or enable science and exploration objectives [1–12]. For example, compared with carrying all of the propellant for a manned round-trip mission to Mars from Earth, producing propellants from Martian CO_2 and hydrogen from Earth can reduce the initial mass launched from low Earth orbit by 20–45%, with even greater leverage when Martian water is used [13]. Sanders et al. point out that there is a significant gap between the analytical studies and the development of the actual ISRU hardware [13], which has resulted in efforts to fill this gap [14–25]. Although the research discussed here focuses on a mission to Mars, much of the technology is directly applicable to lunar resource utilization and habitat air reconditioning.

The Martian atmosphere is the most accessible resource on Mars. It consists primarily of carbon dioxide (95.5%), nitrogen (2.7%), argon (2.6%), and trace other gases, such as water and oxygen, at low pressure (6–10 torr) and low temperature (184–242 K) [4].

Water sources located at the poles, in the soil, and in subterranean reservoirs are a second major resource [3]. Previous researchers have performed several joint and independent studies about ISRU missions [1,5,25]. These studies included modeling ISRU systems for the Mars Human Reference Mission, life support, several Mars robotic missions, and other missions [1,5,25]. Because of the complexity and risk, not to mention the cost, involved in a mission to Mars, it was not prudent to assume that water would be readily available, and/or an astronaut would be on hand to assist the water collection rover(s) or other collection systems should problems occur [i.e., the rover(s) get stuck]. Therefore, the production of fuel and oxidant was to be accomplished using Martian carbon dioxide and Earth-supplied hydrogen [1,5,25].

This process is referred to as in situ propellant production (ISPP). The ISPP integrates CO_2 purifier, concentrator, and compressor, Sabatier reaction (SR), reverse water-gas-shift (RWGS) reaction, and water electrolysis (WE) to make the desired oxidant and fuel. A simplified schematic of the process is in Fig. 1. In this process, CO_2 from the Martian atmosphere is purified, compressed, mixed with H_2, and fed to the RWGS reactor to make CO and water. The CO is discarded, while the water is fed to the WE. The remaining hydrogen and CO_2 are fed to the Sabatier reactor where methane and water are produced. The methane is purified and stored, the water is sent to the WE, and residual CO_2 and H_2 are recycled. For simplicity, the heat exchangers, purifiers, and makeup hydrogen are not shown. Details of the ISPP process can be found in [1,5,13,19,23,25]. Obviously, the hardware must be highly reliable, robust and compact, as well as highly efficient both thermally and electrically.

An analysis of a conceptual proposed mission identified oxygen and methane in a 3.8:1 ratio should be used as the oxidant and fuel, and that approximately 1000 kg of fuel and oxidant was to be a realistic production target. The total production duration was derived from the sample-return mission duration. Orbital mechanics studies have shown that the economics are optimized for on-Mars stays equivalent to 30–90 Earth days [25]. Based on the aforementioned studies and goals, the total production duration was determined by NASA to be 70 days, which translates into approximately 600 g/h production (approximately 128 g CH_4/h and 486 g H_2O/h) [24]. Because mission requirements are constantly being updated, the reactors were designed to meet one-eighth scale mission targets and to show that they can be easily scaled to meet mission requirements. The reactor should maximize carbon dioxide conversion, with the excess hydrogen being recycled into the system. The amount of excess hydrogen will be dependent on the reactor's performance and also the complete ISRU system needs and requirements. This analysis is beyond the scope of this paper, which will solely deal with the reactor performance.

The SR reactor converts the CO_2 to CH_4 and H_2O via exothermic reaction:

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O \quad \Delta H = -165\ \text{kJ/mol} \qquad (1)$$

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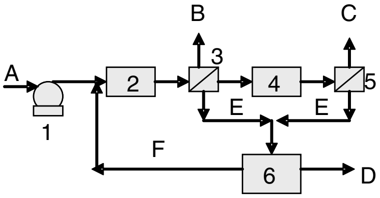

Fig. 1 Simple dchematic of the ISPP process. A = CO_2 from the Martian atmosphere, B = exhaust CO, C = methane to storage, D = oxygen to storage, E = recycled hydrogen, 1 = CO_2 compressor/purifier, 2 = RWGS reactor, 3 = condenser/separator, 4= Sabatier reactor, 5 = condenser/separator, 6 = water electrolysis unit.

Undesirable side reactions [Eqs. (2) and (3)] produce CO in an endothermic reaction and C_2H_6 in an exothermic reaction, which can be rejected from the system as waste products. These reactions should be minimized in the system.

$$CO_2 + H_2 \rightarrow CO + H_2O \quad \Delta H = +46\ \mathrm{kJ/mol} \tag{2}$$

$$CO_2 + 3.5H_2 \rightarrow 1/2C_2H_6 + 2H_2O \quad \Delta H = -132.5\ \mathrm{kJ/mol} \tag{3}$$

The SR reactor is described in [19]. The rest of the water required for the WE unit (approximately 258 g H_2O/h at full scale) to achieve the required oxidant to propellant ratio of 3.8:1 is generated in the RWGS reactor via the endothermic reaction:

$$CO_2 + H_2 \rightarrow CO + H_2O \quad \Delta H = +41\ \mathrm{kJ/mol} \tag{4}$$

The RWGS reaction does compete with the SR reaction, Eq. (1), however, the RWGS is thermodynamically favored at higher temperatures. Although the production of additional methane is not a bad occurrence, to maintain a simple system, the RWGS was operated to minimize the SR reaction.

II. Experimental

The RWGS reactor was designed to operate at high temperatures and with a minimal pressure drop. It was constructed inhouse at Pacific Northwest National Laboratory (PNNL) of inconel with a catalyst bed of active volume of 2.2 cm^3. It was designed for operation at a contact time (CT) of 36 ms. Contact time is the inverse gas hourly space velocity (GHSV) and is based upon the active catalyst bed volume at standard conditions. The reactor was designed to be heated by ten heating rods of 1/8 in. diameter. Thermocouples (type K, accurate to approximately ±5°C) were located at the catalyst bed inlet and outlet to measure the bed temperature, and also in the reactor body at the inlet and middle. Thermocouples were also located at the inlets and outlets of the recuperative heat exchanger. Pressure transducers (0–15 psig) located at the system inlet and outlet were used to measure the entire system drop. Two differential pressure transducers (Sensotec 0–5 psid) were also in the system, one to measure the pressure drop across the reactor itself and another to measure the pressure drop across the reactor and recuperative heat exchanger. A highly efficient recuperative heat exchanger was used to decrease the heat load on the system. The reactor, including catalyst, but not tubing or heaters, had a volume of less than 15 cm^3 and weighed less than 50 g (Fig. 2).

An Ru/ZrO_2-CeO made inhouse was the catalyst used in this work. Characterization of the catalyst is reported in [20]. It was made inhouse with the following procedure.

First, $Ce(NO_3)_2$ (Aldrich) was introduced onto ZrO_2 support by incipient wetness technique. After drying at 110°C for 12 h, the ZrO_2-CeO sample was calcined at 350°C for 3 h. Following that, Ru nitrate (Colonial) was impregnated onto the ZrO_2-CeO. Again, the sample was dried at 110°C in air overnight. Final calcination was carried out at 500°C in air for 3 h.

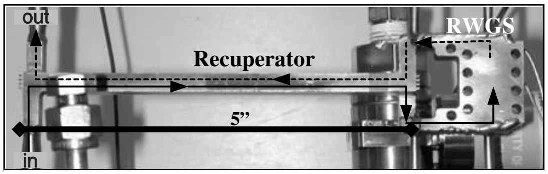

Fig. 2 RWGS reactor and recuperative heat exchanger. Solid lines are reactant feed flow and dotted lines are product flow. Heavy line is for scale.

Structured catalysts were prepared using FeCrAlY intermetallic alloy foam obtained from Porvair. The porosity of the foam is 65 pores per inch. Before the catalyst was wash-coated, the foam was cleaned in ethanol and acetone in an ultrasonicated bath for 20 min. After drying 6 h inside a hood at ambient temperature, the foam was dried at 110°C overnight. The surface of FeCrAlY substrates were heat treated at 900°C in air for 2 h. After heat treatment, the foam substrate was coated with an Al_2O_3 sol gel (SASOL) to further enhance adhesion and exposed surface area. Following the surface treatment, these substrates were wash-coated with catalyst slurry prepared from ball milling of the preceding powder catalysts. To reach desired loading, the wash-coating process was repeated. Between each coating, drying was conducted at 110°C in air. Final calcination was carried out at 400°C for 3 h in air [20].

The catalyst was reduced in the reactor before operation using the following procedure. Under a pure hydrogen flow at 100 standard cubic centimeters per minute (sccm), the temperature was ramped to 400°C at 5°C/ min. After soaking for 2 h, the temperature was increased to desired operation temperature (between 650 and 800°C) still under pure hydrogen. Once the reactor was at the desired temperature, the hydrogen flow was increased to the desired rate and the CO_2 feed was added. For shutdown, the CO_2 feed was shut off first, followed by the hydrogen; a 10% H_2 in He mixture was flowed at 100 sccm over the bed as the reactor was allowed to cool to room temperature.

The reactor test stand is shown in Fig. 3. Brooks mass flow controllers were used for the hydrogen and carbon dioxide. The heat exchanger recovered heat from the product gases to preheat the reactants to near-reacting temperature. The heat exchanger was built using laminate fabrication techniques [26] and had an effectiveness of 0.85–0.9 with low pressure drop, depending on gas throughput. The gases were preheated before entering the recuperative heat exchanger to more accurately simulate their condition in a complete ISPP system (i.e., they will be heated slightly to approximately 100°C due to the compression and purification process described in [25]). Product gases then pass back through the recuperative heat exchanger and into a condenser which removed and collected the water generated. A PermaPure gas diffusion drier was used to further dry the gases. The dry gas flow rate was measured online by a Bios Dry-Cal flowmeter. The gases were analyzed with a dual column

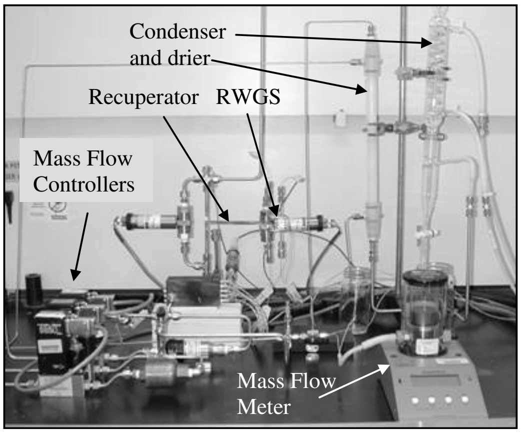

Fig. 3 RWGS test stand.

Table 1 RWGS reactor test conditions

Conditions	Contact time, ms	H_2:CO_2 molar ratio	CO_2 flow, SLM	Temperature, °C
1	36	2:1	1.22	650–800
2	36	1.5:1	1.46	650–800
3	36	1.2:1	1.67	650–800
4	18	2:1	2.44	700–775
5	18	1.5:1	2.93	700–775
6	18	1.2:1	3.33	700–775
7	13	2:1	3.36	700–775
8	13	1.5:1	4.04	700–775
9	13	1.2:1	4.59	750–775
10	8.7	2:1	5.04	775

Agilent MicroGC with Poraplot U and mole sieve column using a thermal conductivity detector.

The RWGS reactor was tested over a variety of temperatures, H_2:CO_2 ratios, and contact times (Table 1). A minimum of three data points were taken at each condition. Carbon dioxide conversion and water generation rate were calculated using the inlet and outlet CO_2 flow rates [in standard liters per minute (SLM)]. The carbon balance typically closed within 1%.

III. Results and Discussion

The RWGS reactor was capable of operating over a wide range of conditions with close to equilibrium performance (Figs. 4–7 and Table 2). At 36 and 18 ms CT, CO_2 conversion was within 95% of equilibrium at close to 800°C operation. Therefore, it was surmised that the heat transport was sufficient for operation at these conditions. At 13 ms CT, the lower H_2:CO_2 ratio of 1.2:1 still achieved near equilibrium operation (within 97% of equilibrium); however, the higher 2:1 ratio flow rates were approximately 90% of equilibrium conversion. The cause for the lower approach to equilibrium could be due to heat transfer limitations, mass transfer limitations, bypass, decreased kinetics due to CO_2 dilution from the high H_2:CO_2 ratio, or, most likely, a combination of these reasons. Because the conversion was high at the lower H_2:CO_2 ratios while at the same CT, it is not likely that bypass is a significant factor. It is possible that there is a mass transfer limitation. The high amount of hydrogen is interfering with the carbon dioxide diffusing to the catalyst surface, as well as decreasing the kinetics. For the 36 ms CT operation, the higher 2:1 ratio operation was closest to equilibrium. However, at the

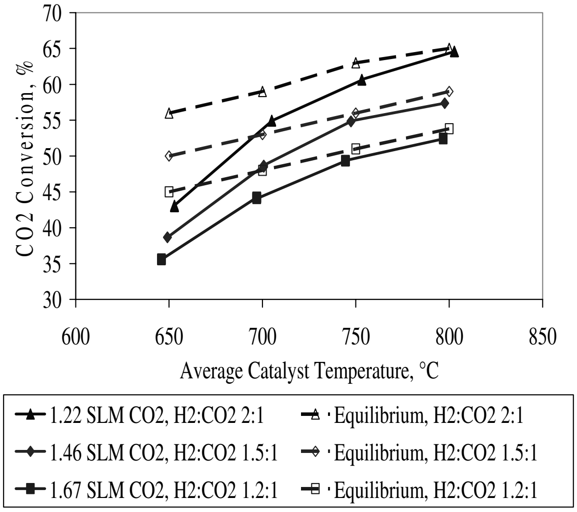

Fig. 4 Carbon dioxide conversion at 36 ms CT (GHSV = 100,000 h^{-1}) and atmospheric pressure. There was 100% selectivity to CO. The reactor pressure drop was 1.6–2.2 kPa. The reactor plus recuperator pressure drop was 9–11 kPa.

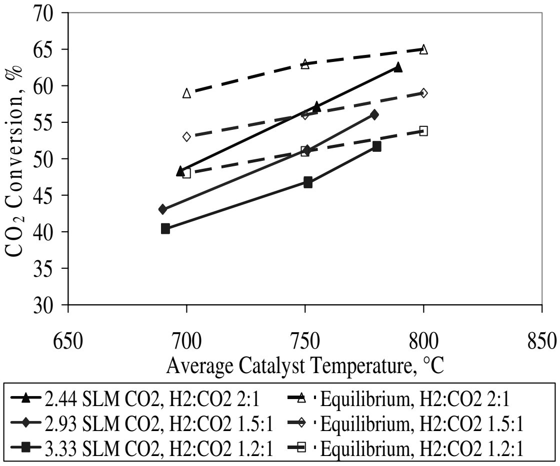

Fig. 5 Carbon dioxide conversion at 18 ms CT (GHSV = 200,000 h^{-1}) and atmospheric pressure. There was 100% selectivity to CO. The reactor pressure drop was 3.5–4.2 kPa. The reactor plus recuperator pressure drop was 21–23 kPa.

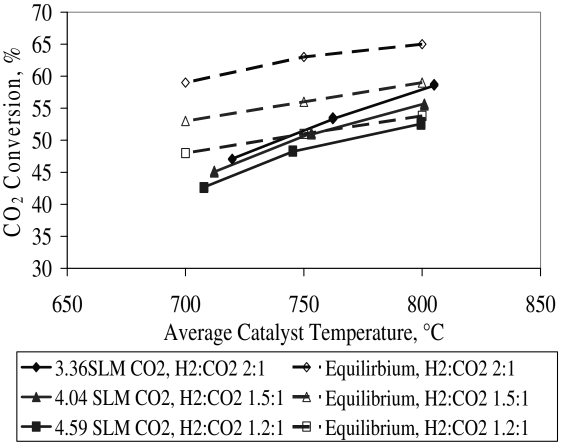

Fig. 6 Carbon dioxide conversion at 13 ms CT (GHSV = 277,000 h^{-1}) and atmospheric pressure. There was 100% selectivity to CO. The reactor pressure drop was 5–6 kPa. The reactor plus recuperator pressure drop was 37–45 kPa.

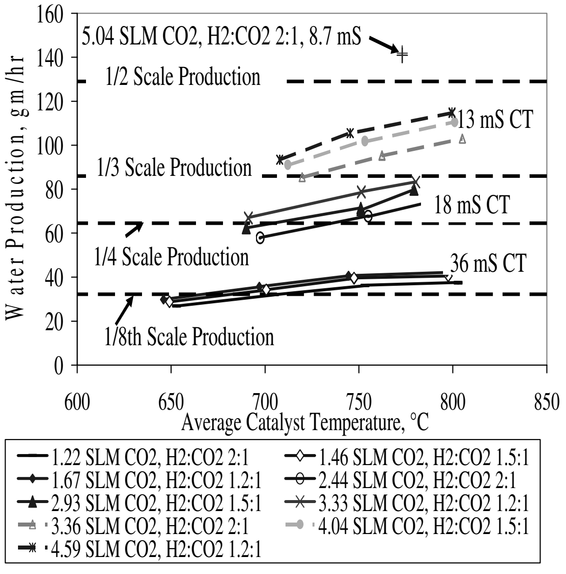

Fig. 7 Water production.

Table 2 RWGS reactor testing results

Test	Contact time, ms	H_2:CO_2, molar	CO_2 flow, SLM	T, °C	Conv. %	CO_2 reacted, SLM	H_2O prod. g/h[a]	Specific productivity,[b] h^{-1}	Pressure drop,[c] kPa, psi
1	36	2	1.22	653	43.0	0.525	26.9	0.54	1.6 (0.22)
2	36	2	1.22	705	54.9	0.670	32.0	0.64	1.7 (0.24)
3	36	2	1.22	753	60.6	0.739	36.2	0.724	1.8 (0.26)
4	36	2	1.22	803	64.6	0.788	37.4	0.75	2.0 (0.30)
5	36	1.5	1.46	649	38.7	0.565	28.8	0.576	1.7 (0.25)
6	36	1.5	1.46	700	48.6	0.710	34.2	0.684	1.7 (0.25)
7	36	1.5	1.46	747	54.8	0.800	39.5	0.79	1.9 (0.27)
8	36	1.5	1.46	798	57.4	0.838	40.5	0.81	2.1 (0.31)
9	36	1.2	1.67	646	35.6	0.594	29.8	0.596	1.7 (0.25)
10	36	1.2	1.67	697	44.1	0.736	35.6	0.712	1.8 (0.26)
11	36	1.2	1.67	744	49.3	0.823	40.6	0.812	2.0 (0.29)
12	36	1.2	1.67	797	52.4	0.875	42.3	0.846	2.2 (0.32)
13	18	2.0	2.44	697	48.3	1.18	57.9	1.16	3.5 (0.5)
14	18	2.0	2.44	755	57.2	1.40	67.7	1.35	3.7 (0.54)
15	18	2.0	2.44	789	62.6	1.53	74.6	1.49	4.0 (0.58)
16	18	1.5	2.93	690	43.1	1.26	62.3	1.25	3.6 (0.53)
17	18	1.5	2.93	751	51.2	1.50	71.1	1.42	3.8 (0.55)
18	18	1.5	2.93	780	56.0	1.64	79.9	1.60	4.1 (0.60)
19	18	1.2	3.33	691	40.4	1.35	66.9	1.34	3.7 (0.54)
20	18	1.2	3.33	751	46.8	1.56	78.7	1.57	4.0 (0.58)
21	18	1.2	3.33	780	51.7	1.72	83.2	1.66	4.2 (0.61)
22	13	2.0	3.36	719	50.6	1.70	85.4	1.71	5.0 (0.73)
23	13	2.0	3.36	762	57.9	1.95	95.3	1.91	5.4 (0.78)
24	13	2.0	3.36	805	62.8	2.11	103.0	2.06	5.7 (0.83)
25	13	1.5	4.04	712	45.8	1.85	91.1	1.82	5.2 (0.72)
26	13	1.5	4.04	753	52.0	2.10	102.0	2.04	5.5 (0.80)
27	13	1.5	4.04	801	57.0	2.3	110.5	2.21	5.8 (0.84)
28	13	1.2	4.59	708	42.1	1.93	93.4	1.87	5.3 (0.77)
29	13	1.2	4.59	745	47.7	2.19	105.5	2.11	5.7 (0.83)
30	13	1.2	4.59	800	52.6	2.41	114.8	2.30	6.0 (0.87)
31	8.7	2.0	5.04	773	57.95	2.92	154.1	3.08	7.6 (1.10)

[a] Water production based upon amount of CO_2 converted and assumes 100% recovery.

[b] Specific productivity is the water production per hour (g/h) divided by the reactor mass (50 g).

[c] Pressure drop for the reactor only.

lower CTs, the higher ratio operation was the furthest from equilibrium. This seems to support this hypothesis. Also, there may have been insufficient heat transfer into the reactor at the higher ratios due to the increased amount of products produced, which increased the heating load. Also, the heat transfer rate would be lower at the 2:1 ratio feed flow compared with that of the 1.2:1 ratio stream due to a decrease in the thermal capacity (38.7 J/mol · K and 41.7 J/mol · K, respectively). Most likely, it is a combination of the heat and mass transfer limitations.

The reactor's performance was better than the targeted design points. A maximum water production of 154.1 g H_2O/h was achieved, which is above half-scale production for the mission. Because the mass in an extraterrestrial system is so important, the production was normalized by dividing the water generation rate by the RWGS reactor mass. This translates into a specific water production of 3.08 g H_2O/(h · g reactor) or 10.3 g H_2O/(h · cc reactor). Methane production was less than detectable limits (approximately 0.01%) resulting in a selectivity to carbon monoxide of >99.99%. Even though a lifetime test of the reactor was not conducted, it was operated over 35 h with six thermal cycles without any noticeable deactivation.

Because the Martian atmosphere is at such a low pressure, the system operating pressure is limited by the CO_2 compressor. To minimize the electrical parasitic power, low-pressure operation is desirable because less compression, and therefore less power, is required. Therefore, the reactor pressure drop is extremely important and was designed to be less than 4 kPa at a CT of 36 ms. The measured pressure drop was 1.6 kPa at 36 ms CT, which was significantly less than anticipated. The maximum pressure drop was 7.6 kPa, but this was achieved at a CT of 8.7 ms, over four times the designed operation. This better-than-anticipated performance was due to the impregnation of the foam with the catalyst not occupying as much space as anticipated, and therefore resulting in less pressure drop than expected.

This high performance with minimal pressure drop supports the development and use of microreactors for ISPP applications. Depending on how the reactor is operated, two to four of these reactors would be able to meet the mission goals for water production.

IV. Conclusions

A reverse water-gas-shift reactor was developed and demonstrated for use in an ISPP mission. The reactor was originally designed to produce 32.25 g H_2O/h (one-eighth the targeted mission requirements), but was able to achieve significantly higher results: up to 129 g H_2O/h (one-half the mission target). When normalized by weight, this result was over 3.08 g H_2O/h per gram of reactor mass. The compact (<15 cm^3) and lightweight (<50 m) reactor had a low pressure drop of 1.6 kPa at the design operating conditions (36 ms CT). At the higher flow rates of 8.7 ms CT, the pressure drop was still reasonable at 7.6 kPa. The mission target can be achieved by using two to four of these units.

Acknowledgment

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