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#### 1 Introduction

Solar-made zinc is an attractive renewable energy carrier [1]. It is a compact solid fuel that finds applications in Zn/air fuel cells and batteries. Zinc can also be reacted with water in an exothermic reaction to form high-purity hydrogen [2-5]. In either case, the chemical product from these power generation processes is ZnO, which in turn is recycled and solar-reduced to Zn. In recent reviews, the two-step H<sub>2</sub>O-splitting solar thermochemical cycle based on ZnO/Zn redox reactions has been identified as a promising path for solar hydrogen production [6,7]. The thermal dissociation of ZnO into its elements requires temperatures above 2000 K, and the product gases need to be either quenched or separated to prevent their recombination [2,8,9]. The reaction temperature can be significantly lowered and the recombination avoided by using carbonaceous materials as reducing agents. In the presence of reducing agents such as coal, coke, biomass, and methane, the uptake of oxygen by the formation of CO makes the reduction feasible at 1300-1700 K [10] and allows the use of

# A 300 kW Solar Chemical Pilot Plant for the Carbothermic Production of Zinc

In the framework of the EU-project SOLZINC, a 300-kW solar chemical pilot plant for the production of zinc by carbothermic reduction of ZnO was experimentally demonstrated in a beam-down solar tower concentrating facility of Cassegrain optical configuration. The solar chemical reactor, featuring two cavities, of which the upper one is functioning as the solar absorber and the lower one as the reaction chamber containing a ZnO/C packed bed, was batch-operated in the 1300–1500 K range and yielded 50 kg/h of 95%-purity Zn. The measured energy conversion efficiency, i.e., the ratio of the reaction enthalpy change to the solar power input, was 30%. Zinc finds application as a fuel for Zn/air batteries and fuel cells, and can also react with water to form high-purity hydrogen. In either case, the chemical product is ZnO, which in turn is solar-recycled to Zn. The SOLZINC process provides an efficient thermochemical route for the storage and transportation of solar energy in the form of solar fuels. [DOI: 10.1115/1.2711471]

Keywords: solar energy, renewable energy, hydrogen, zinc, solar electricity, solar chemistry, solar energy storage

conventional Zn condensation technologies [11]. The solar carbothermic reduction of ZnO has been experimentally demonstrated using C(s) [12–15] and CH<sub>4</sub> [16,17] as reductants. From the standpoint of CO<sub>2</sub> avoidance, zinc production by solar-driven carbothermic reduction of ZnO offers a CO<sub>2</sub> emission reduction by a factor of 5 vis-à-vis the conventional fossil-fuel-driven electrolytic or Imperial Smelting processes [10]. Obviously, if biomass is used as a reducing agent, it becomes a zero-net CO<sub>2</sub> process. The cyclic process from solar energy to electricity via solar-processed Zn/air fuel cells is being investigated within the framework of the European Union's project SOLZINC [18].

The carbothermic reduction of ZnO is complex, but the overall reaction can be represented by

$$ZnO + C \rightarrow Zn(g) + CO$$
 (1)

This reaction proceeds endothermally  $(\Delta H_{1500 \text{ K}}^2=350 \text{ kJ/mol})$  at temperatures above 1200 K. Its rate depends strongly on the carbon source [15,19–21]. The advantages of using solid carbon-aceous materials as reducing agents, as compared to gaseous hydrocarbons, include their more versatile availability, the option of using biomass, and the higher fraction of solar power transferred to the product Zn rather than the by-product off-gas [10]. Various solar reactor concepts for effecting reaction (1) have been proposed and were tested on laboratory scale [13–15,19,22].

In previous papers, we described in detail the design, modeling,

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Contributed by the Solar Energy Engineering Division of ASME for publication in the JOURNAL OF SOLAR ENERGY ENGINEERING. Manuscript received January 10, 2006; final manuscript received March 29, 2006. Review conducted by Robert Pitz-Paal. Paper presented at the 2005 International Solar Energy Conference (ISEC2005), August 6–12, 2005, Orlando, Florida.



Fig. 1 Flow diagram of the SOLZINC solar chemical pilot plant featuring the solar concentrating system, the solar reactor, the off-gas system, and the diagnostic instrumentation

fabrication, and experimental investigation of a 5-kW solar chemical reactor for performing the carbothermic reduction of ZnO [15,19,20,23–27]. The reactor concept was based in the so-called "two-cavity" reactor, and has been selected for up-scaling to 300 kW in the SOLZINC project. This paper describes its engineering design and its integration in the solar chemical plant, and presents the experimental investigation of the reactor's performance. Problems associated with the operation of the reactor and its peripheral components are discussed.

#### 2 Engineering Design

The main components of the SOLZINC chemical pilot plant are depicted in Fig. 1; namely, the solar concentrating system, the solar reactor, the off-gas handling system, and the diagnostics instrumentation.

**2.1** Solar Concentrating System. The "beam-down" Cassegrain optical configuration of the Weizmann Institute of Science's (WIS's) solar tower consists of a field of heliostats (two-axis tracking parabolic mirrors) that focus the sun's rays onto a hyperbolical reflector at the top of the tower to re-direct sunlight to a compound parabolic concentrator (CPC) located on the ground level [28]. Figure 2 shows a photograph of the facility and indicates the optical path. The truncated 3D-CPC, with a 220-cm-diameter entrance, 46-cm-diameter exit, and 500-cm height, delivers about 300 kW of concentrated solar power with an exit angle of about 140 deg. The exit of the CPC is close-coupled to the reactor's aperture.

**2.2** Solar Reactor. The schematic of the two-cavity solar reactor is shown in Fig. 3. It features two cavities in series, with the upper cavity functioning as the solar absorber, and the lower cavity as the reaction chamber. With this arrangement, the upper cavity protects the window against particles and condensable gases and further serves as a thermal shock absorber. The upper cavity comprises a 48-cm-diameter aperture with a 60-cm-diameter 12-mm-thick quartz window mounted on a water-cooled copper ring, a 0.8-cm-thick graphite or SiC/graphite partition separating

the two cavities, and inlet/outlet ports for an inert window-cooling carrier gas. The lower, 140-cm-diameter 0.8-m-high cylindrical cavity contains the packed bed of ZnO-C mixture. The maximal initial height of the batch is 0.5 m, corresponding to about 500 kg of input material and allowing for a full day operation, so that cooling and recharging of a new batch can be accomplished overnight. For this purpose, the lower cavity can be easily disconnected for refilling. Both upper and lower cavities have a steel outer shell, followed by a 1-cm-thick foam insulation ( $\lambda = 0.03 \text{ W/m/K}$ ), a 17.5-cm-thick Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> lining ( $\lambda = 0.3 \text{ W/m/K}$ ), and 0.8-cm-thick SiC plates. The SiC inlet pipe carries inert gas or recycled off-gas to sweep product gases. The



Fig. 2 The "beam-down" Cassegrain optical configuration of the WIS's solar tower concentrating system, consisting of a field of heliostats that focus the sun rays onto a hyperbolical reflector at the top of the tower to re-direct sunlight to a CPC located on the ground level [28]

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Fig. 3 Schematic of the solar chemical reactor, featuring two cavities in series with the upper one functioning as the solar absorber and the lower one as the reaction chamber containing a ZnO/C packed bed

window in the upper cavity is flushed with an inert gas flow (FUG in Fig. 1), which leaves through small gaps in the partition plates between upper and lower cavities. The SiC offgas pipe is electrically heated to prevent Zn condensation and extends into the quenching section. An adjustable pressure safety valve ensures that the difference between reactor and ambient pressures does not exceed 10 kPa, the maximum allowed for the flat quartz window. Generic design guidelines for such a two-cavity reactor concept have been described previously [23–27], and include the effect of a selective window on the cavities' temperatures, and a numerical reactor model describing the heat and mass transfer phenomena occurring in the shrinking packed bed.

**2.3 Off-Gas Handling System.** Figure 4 shows a photograph of the solar reactor and the off-gas quenching and treatment system installed next to the exit of the reactor. Product gases exiting the reactor are quenched in a vertical cooler #1 with recycled cold off-gas (indicated by the inner recycling loop in Fig. 1), resulting in the formation of Zn particles with a typical mean size of  $3-5 \ \mu$ m, which are separated in a cyclone. Fines are further



Fig. 4 Photograph of the 300-kW solar reactor and its peripheral components, installed under the solar tower beam-down concentrating system

transported by a fan and recycled to the quenching chamber for further growth. The fan is frequency controlled and runs at a rate complying with the desired reactor pressure in the range 0-0.5 kPa above ambient. A gas/particle fraction leaves this inner loop and the fines are filtered downstream. The cleaned off-gas, mainly CO, is partially recycled into the reactor and serves as carrier gas (indicated by the outer recycling loop in Fig. 1), while the remaining leaves through a chimney. In an industrial plant, its energy content would be used, e.g., to drive a heat engine or the CO could be water-shifted for hydrogen production. As far as the balance of the plant is concerned, cooling water is employed for the Cu-mounting of the quartz window, for the walls of coolers #1 and #2, and for the walls of the CPC; nitrogen is employed for flushing the quartz window from the inside, for supplying further carrier gas that sweeps product gases (optionally, as this can be substituted by off-gas from the outer recycling loop), and for purging the pressure sensor pipes in the dusty section of the plant.

**2.4 Diagnostic Instrumentation.** More than 150 sensors monitor and control the process. Thermocouples type-N are used for measuring the reactor outer wall temperatures, and type-B in an alumina sheath for the reactor inner wall temperatures. Critical are the temperature above the packed bed (TLP1) and the temperature of the partition wall (TRP2), since these are representative for the lower and upper cavity, respectively. The thermocouples in the off-gas system are fastened on the metal pipes. Pressure sensors monitor the reactor and off-gas system pressures. Gas composition is measured every second, using IR for CO,  $CO_2$ , paramagnetism for  $O_2$ , and thermal conductivity for H<sub>2</sub>.

#### **3** Experimental Results

3.1 Test Procedure. Batches of industrial ZnO powder (Grillo 2011) and industrial beech charcoal powder (Chemviron) with a typical C<sub>fix</sub>:ZnO molar stoichiometry of  $\alpha = 0.9$ :1 were prepared in an industrial batch mixer (Loedige FKM 300 D). A 116-kg mixture was loaded uniformly in the lower cavity, resulting in a 12-13 cm thick porous bed. Prior to the start, the whole system was flushed with N2, until the O2 concentration was below 1 vol.- %. Heating of the solar reactor to the desired temperature was normally performed at a rate of about 10 K/min by successively introducing heliostats. The frequency of the fan and the position of valve V6 in the inner recycling loop (see Fig. 1) were adjusted to restrict the difference between the reactor and ambient pressures DPL to 0.5 kPa, and to operate at a set inner recycling flow rate FQ2. Baseline operation conditions were: a temperature set point of the offgas pipe electric heater of 1200 K, N<sub>2</sub> flow rate into the upper chamber of 6 Nm<sup>3</sup>/h, N<sub>2</sub> flow rate into the lower chamber in the range of 3-6 Nm<sup>3</sup>/h, gas mass flow rate in the outer recycling loop of 0-20 Nm3/h, and a flow rate of the quenching gas FQ2 of 350-500 Nm<sup>3</sup>/h.

**3.2 Experimental Results.** Table 1 summarizes the operational parameters and test results of 5 selected experimental runs A–E. Run C is presented in more detail in Figs. 5–7 and 9. Batches of 1.23 kmol ZnO mixed with beech charcoal were used according to stoichiometric ratio  $\alpha$ .

The time variation of the major temperature readings of the reactor and off-gas system during the experimental run C are shown in Figs. 5 and 6, respectively. Highest temperature of 1550 K was obtained on the partition wall (TRP2), while the temperature in the upper part of the reaction chamber was about 50-100 K lower, as predicted by radiation heat transfer modeling [23]. The temperature of the SiC floor of the reactor (TLP9) indicated a high-temperature gradient through the packed bed. As the bed shrinked, it approached the value of the lateral wall temperature. Figure 6 demonstrates the effective cooling of the offgas in the system. Specifically, the temperature TQG3 after cooler #1 was below the melting point of Zn (692 K).

Figure 7 shows the offgas composition and mass flow rate for

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Experimental run	А	В	С	D	Е
Stochiometry $\alpha = C_{fix}/ZnO$	0.9	0.9	0.9	1	0.8
$N_2$ mass flow rate upper chamber	6	6	6	6	6
FUG [Nm <sup>3</sup> /h]					
$N_2$ mass flow rate lower chamber	3	3	3–9	3–9	3–9
FLG [Nm <sup>3</sup> /h]					
Outer recycl. gas flow rate FQ1	0–6	16	0-10	0-10	0-10
$[Nm^3/h]$					
Inner recycl. gas flow rate FQ2, Nm <sup>3</sup> /h	500	500	350	350	350
А	t maximal pow	er input			
$T_{1}$ = TLP1 [K]	1440	1390	1450	1460	1470
$\dot{m}_{7}$ [kg/h]	42	29	44	46	40
$\dot{m}_{\rm Zn}$ [kmol/h]	0.64	0.44	0.67	0.7	0.61
Inte	egrated over run	n duration			
$n_{\rm O}$ [kmol]	0.81	1.55	1.15	1.25	1.14
n <sub>c</sub> [kmol]	0.71	1.16	1.03	1.13	0.96
$n_c/\alpha$ [kmol]	0.79	1.29	1.15	1.13	1.2
Cyclone location after cooler Nr.	2	2	1	1	1
m <sub>cyclone</sub> [kg]	13.5	34	53	45.5	44.5
m <sub>cvclone</sub> [kmol]	0.21	0.52	0.81	0.7	0.68
Batch height after test [cm]	4	<1	<1	<1	<1

Table 1 Major operational parameters and test results of selected pilot plant experiments

the experimental run C, averaged over 2.5 min. The off-gas flow rate data fluctuated by some  $\pm 10\%$  due to short N<sub>2</sub> pulses introduced in an off-gas pipe bend to avoid clogging. Once the temperature at the packed-bed surface exceeded 800 K, beech charcoal pyrolysis started releasing mainly CO<sub>2</sub> and H<sub>2</sub>. At above 1300 K, the carbothermic reduction of ZnO occurred and CO became the predominant component of the off-gas. The Zn production rate was calculated from the oxygen balance

$$\dot{n}_{\rm Zp} = \dot{n}_{\rm O} = \dot{n}_{\rm CO} + 2\dot{n}_{\rm CO_2} \tag{2}$$

where  $\dot{n}_i$  is the molar flow rate of species *i*. This rate agreed well with the respective estimate of the reaction rate based on the C-balance

$$\dot{n}_{\rm Zn} = \dot{n}_{\rm C}/\alpha = (\dot{n}_{\rm CO} + \dot{n}_{\rm CO_2})/\alpha \tag{3}$$



Fig. 5 Variation of the solar reactor temperatures during the solar experimental run C. TLP1–8 are located in the lower cavity at different heights, every 10 cm (TLP1 is the top temperature, TLP9 is the bottom temperature)

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Fig. 6 Variation of the offgas system temperatures during the solar experimental run C. The location of thermocouples is indicated in Fig. 1

The total amount of Zn produced, calculated by integration of the Zn production rate over the duration of the experimental run, was consistent with the amount of reactants consumed. Some tests experienced a surplus of oxygen, sucked from ambient air into the under-pressure regions of the off-gas system. A secondary reaction of  $CO_2$  with the graphite separation wall plates resulted in a weight loss equivalent to 3% of the total carbon in the batch and, hence, negligible for the carbon balance. This corrosion may be further reduced by using separation plates from SiC-graphite composites, as demonstrated in laboratory scale experiments [20].

The dependence of the reaction rate on temperature is shown in the Arrhenius plot of Fig. 8, where TLP1 was used as the reference temperature of the lower cavity. Each point in the diagram corresponds to the quasi-stationary region of an operational day. The rates are comparable for batches with a stoichiometry of  $\alpha$ =1 and 0.9. Run E for  $\alpha$ =0.8 resulted in a slightly lower reaction rate, in agreement with lab-scale experiments with the 5-kW reactor prototype [15,20] and with modeling predictions [27]. Variations of FLG+FQ1 and of the type of carrier gas (N<sub>2</sub> or recycled off-gas) did not affect significantly the reaction rate, similar to the observations in the lab-scale experiments [20]. As predicted by heat and mass transfer analysis, the large temperature gradient within a few millimeters of the top layer is typical of ablation processes where heat transfer through the bed becomes the rate controlling mechanism [26,27].

3.3 Product Characterization. X-ray diffraction (XRD) analysis of samples collected in the cyclone and the bag filter indicated a Zn purity of around 95% (some 5% ZnO). Samples from deposits in the pipes have similar purity. A ZnO content of a few mol % is advantageous, since the respective ZnO layer reduces the reactivity of the Zn dust to a level desired for its application in Zn/air batteries and it is also standard in conventional commercial Zn dust production. Figure 9 shows the size distribution of particles separated by the cyclone and by the bag filter for run C. As expected, the bag filter dust is finer than the cyclone dust. Fifty percent of the particles are smaller than 2.5 and 5  $\mu$ m for the bag filter and cyclone dusts, respectively. The reactivity of the zinc dust with air was examined in separate laboratory tests and found to be suitable for anodes in Zn/air fuel cells. If Zn is further processed to H<sub>2</sub> via steam-hydrolysis, the off-gas system may need to be adjusted to produce Zn nanoparticles [4,5] or a Zn melt [3].

**3.4 Efficiencies.** Two energy conversion efficiencies are used to describe the thermal performance of the solar pilot plant:

(1) The *thermal efficiency* of the solar reactor is defined as the fraction of solar power input entering through the reactor's aperture that is net absorbed as sensible and process heat (i.e., reaction enthalpy)



Fig. 7 Off-gas flow rate, off-gas composition, and Zn production rate, as a function of time during the experimental run C of Table 1

$$\eta_{\rm thermal} = \frac{\Delta H}{Q_{\rm solar}} \tag{4}$$

(2) The *process efficiency* is defined as the ratio of the heating values of Zn and CO produced to the thermal energy input (i.e., solar energy input and heating value of the carbon consumed)

$$\eta_{\text{process}} = \frac{HV_{\text{Zn}} + HV_{\text{CO}}}{Q_{\text{solar}} + HV_{\text{C}}}$$
(5)

 $Q_{\text{solar}}$  is calculated by a ray-tracing program that takes into account the individual heliostats used as a function of time, and was validated by calorimetric tests. For run A,  $Q_{\text{solar}} \approx 280 \text{ kW}$ ,  $HV_{Zn}$ =63 kW,  $HV_{CO}$ =36 kW,  $HV_{C}$ =60 kW,  $\eta_{\text{thermal}} \approx 30\%$ , and  $\eta_{\text{process}} \approx 30\%$ . Losses are due mainly to reflection at the quartz window, re-radiation through the aperture, heat conduction through the walls, and transients. In addition, some power is required for auxiliary components, especially for the main off-gas fan, with a rating of 4 kW. The off-gas pipe heater is typically only operating during heat-up and cool-down phases. Significantly higher efficiencies are expected for larger, industrial plants because of the improved reaction surface area-to-wall area ratio. An additional efficiency increase and, consequently, cost reduction, could be realized by eliminating the quartz window, provided suitable partition wall materials are identified that can operate gastight and thermal-shock resistant at 1500 K in air. For industrial operation, the energy required for heating the reactor to a quasistationary temperature profile may be delivered during the morn-



Fig. 8 Arrhenius plot for zinc production rate in quasistationary operation periods as a function of the temperature TLP1 (wall temperature in upper part of lower cavity)

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Fig. 9 Particle size distribution of Zn collected in cyclone and bag filter during test C. Shown is the volume fraction of all particles below a certain particle size

ing hours of low irradiation intensity. The approximate thermal mass of the reactor is 150 kWh, corresponding to about 30 min heating. Recovery of the sensible/latent heat of the products may further increase the process efficiency. The overall solar-to-fuel process efficiency of a solar plant should also account for the optical efficiency of the solar concentration system, i.e., the portion of solar power impinging on the heliostats surfaces that enters the solar receiver-reactor, estimated at 55–65% [29].

3.5 Practical Problems Encountered During Operation. The CPC and its coupling with the quartz window performed well. After the first solar test with a ZnO-C batch, Zn droplets were found on cold parts of the upper cavity, indicating that Zn vapor penetrated the upper cavity through small gaps of the partition wall. This problem was eliminated by minimizing the gaps with small graphite plates and by increasing the N<sub>2</sub> purging flow. After each experimental run, the quartz window was covered by a thin dust layer that could be easily removed without the need of dismounting the window. After several heating-cooling cycles, cracks were observed in the SiC-lining of the upper cavity. SiC cement showed better thermal stability. Cracks were also observed in the insulation material in the upper cavity due to thermal expansion/contraction. Filling the empty spaces with soft insulation material and closing with Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> cement solved this problem. Zn vapor condensed and deposited in the off-gas pipe before cooler #1 (see Figs. 1 and 4), where it was partially oxidized to ZnO. Deposition was significantly reduced by increasing the length of the electrical off-gas pipe heater to keep temperatures above the Zn(g) saturation. The Zn-dust cyclone separator worked well and the fraction separated increased after changing its location to follow cooler #1 instead of cooler #2 (compare Table 1).

#### 4 Summary and Conclusions

A 300-kW solar chemical pilot plant for the production of Zn by carbothermic reduction of ZnO has been successfully commissioned and operated. Main components are the beam-down solar concentrator system, the two-cavity solar reactor, and the off-gas quenching/recycling unit. The solar chemical reactor, containing a packed-bed of ZnO and C, was batch-operated in the 1300–1500 K range at conditions that are typical of ablation processes, where heat transfer through the bed becomes the rate controlling mechanism. The Zn production rate was 50 kg/h, with a purity of 95% and a mean particle size in the range  $2.5-5 \ \mu$ m. The thermal efficiency (ratio of reaction enthalpy to solar power input) and the process efficiency (ratio of heating values of products to the thermal energy input) reached 30%.

The experimental demonstration of the solar chemical pilot plant at a power level of 300 kW marks an important step on the path to commercialization of the technology. Potential applications of solar Zn include its use as energy carrier in cyclic processes for on-demand solar electricity and solar hydrogen production, as well as its use as material commodity.

#### Acknowledgment

Funding by the European Community's Research Directorate (Contract ENK6-CT2001-00512) and by the Swiss State Secretariat for Education and Research (SER) is gratefully acknowledged. We thank A. Segal for performing ray tracing calculations for the solar power input into the reactor and A. Frei for XRD analyses of product samples.

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