Master Thesis Description

Title
Surface Temperature of Reacting Powders in High Temperature Solar Chemistry

Short Abstract
Develop a photometric method to quantify the dynamic optical properties of directly irradiated and reacting samples in solar simulators. Linking optical properties to photometric data will enable in-situ assessment of emissivity and temperature, advancing the study of solar-driven reaction kinetics.

Keywords
Concentrated solar power, high flux solar simulator, solar furnace, reactor technology, temperature measurement, pyrometry, infrared radiation, solar-blind optical techniques, photometric data acquisition, optics, reflectivity, emissivity, thermogravimetric kinetic analysis, reaction kinetics.

Description
Solar thermochemical H2O and CO2 splitting is considered as a pathway towards large-scale production of clean and renewable synthetic fuels. A scale-up 100-kWth reactor for the thermal dissociation of ZnO into metallic Zn has been designed, constructed, modeled and successfully tested. Solar-thermogravimetric (solar-TG) analysis is instrumental in the study of the reaction kinetics of the directly irradiated materials in such reactors. Temperature quantification of the reactants surface is a critical input for solar-TG analysis that cannot be attained by thermocouples. Therefore, surface temperature has to be determined optically by use of solar-blind pyrometry. However, its accuracy depends on knowledge of the thermo-optical properties of the sample (i.e. emissivity), which are rapidly changing during experimentation (Figure 1). The objective of the project is to develop a photometric method to quantify the dynamic emissivity of directly irradiated and reacting samples, enabling reliable surface temperature measurement. By use of in-situ experimental tools (thermocouple, calibrated pyrometer, CCD camera), it will establish a link between photometric data (pixel intensities) and emissivity (Figure 2). During experimentation, any observed change in photometric data is the convolved effect of changes in temperature, background radiation and surface morphology that affects emissivity. The thermocouple data will allow for a first crude temperature estimate. Temperature gradients between the sample surface and the thermocouple position introduce a significant temperature error. These gradients depend on porosity and other material properties and may be as high as 500 K over a distance of 5 mm. These errors have to be quantified. After accounting for the change in temperature, the remaining unknown quantities cannot be deconvolved in order to isolate emissivity. Moreover, emissivity anisotropy that may occur during chemical reaction invalidates the assumption of a lambertian (diffusely reflecting) target and necessitates the determination of directional emissivity which can be calculated by obtaining in situ measurements of the bidirectional reflectivity distribution function (BRDF). To this end, an in situ

reflectivity measurement module has to be designed, implemented and added to the setup to obtain reflectivity values as a function of incidence angle.

The reflectometer will consist of a laser mounted on a rail that will be motion controlled by two motors moving along a spherical arc (meridian) with the sample at the center of the sphere. With the laser beam pointing towards the sample from various angles along the arc, the reflected part of the incident laser beam will be detected by the CCD camera allowing for angularly resolved reflectivity values. Such a motorized setup, combined with a CCD camera readout of 1s will enable a sampling period of 30s to keep track of the various dynamic quantities.

To detect the reflected laser radiation in the presence of the intense background radiance (from the Xenon arc lamps of the simulator), the camera sensitivity has to be restricted in a narrow spectral band around the laser wavelength by use of a narrow bandwidth transmission filter. Furthermore, the operating wavelength of the laser and filter can be selected to fall within one of the minima of the Xenon-arc spectrum to further suppress the unwanted radiation.

Prerequisites

An extensive experimental study of the thermo-optical properties has to be conducted and the motorized laser reflectometer has to be implemented. Therefore, we are looking for a self-directed, highly motivated student with experimental skills, an inclination for hands-on lab work and ideally with a mechanical engineering background.

Goal

Develop a photometric method to quantify the dynamic optical properties of directly irradiated reacting samples.

1. Literature review of reactor technology, solar-blind pyrometry, photometric data acquisition reflectivity measurements, solar-TG.
2. Lab introduction, safety briefing and Training in working with dangerous radiation sources (lasers, high-flux solar simulators).
3. Proof of concept experiments in the small scale optical lab
4. Design, implementation and setup of the laser-reflectometer in the solar TG
5. Selection of the laser operating wavelength so that it matches the minimum of the parasitic radiation.
6. Selection of a narrow bandwidth transmission filter to match the camera's sensitivity to the laser's operating wavelength.
7. First, the setup will be used to measure the high-temperature emissivity of well-behaved non-reacting non-evaporating samples such as ceria powder and Zirconia.
8. The method will be tested on Zirconium that undergoes a phase change at a known temperature of 2128K [2]. Associating the known melting temperature with the apparent temperature measured with the pyrometer will allow to independently calculate emissivity and compare the result with the one obtained with our setup. These experiments will test the validity and possible limitations of the approach.
9. The method will be put to a final test by measuring the dynamic high-temperature emissivity during thermal dissociation of ZnO powder.
10. Establish a link between emissivity and photometric data.
11. Evaluate the accuracy of the method under a range of experimental conditions and materials.

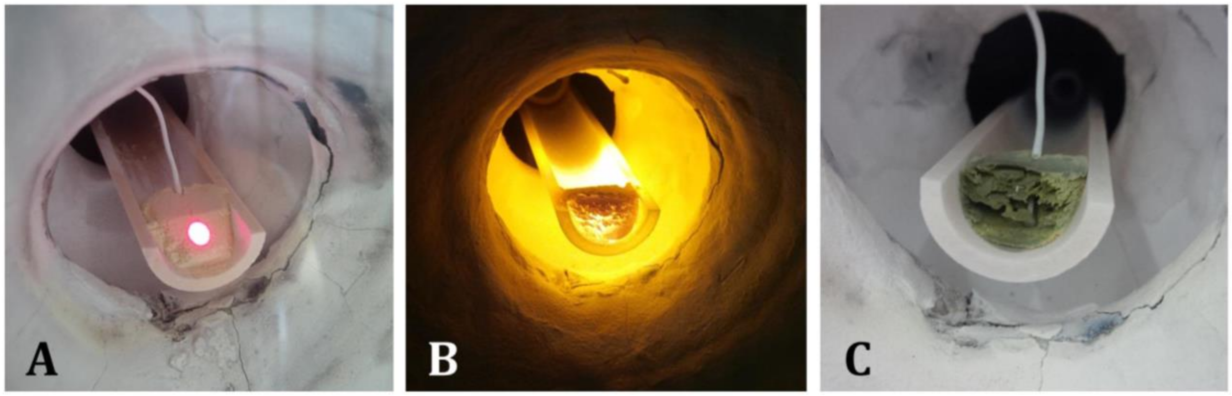

Figure 1. : (A) Sample material in the solar-TG and aligned with a pyrometer and thermocouple. (B) Sample after 120s of high temperature sintering and dissociation. (C) Final sample image after cooling. Material is darkened, hard-sintered, and displaying unique surface morphology.

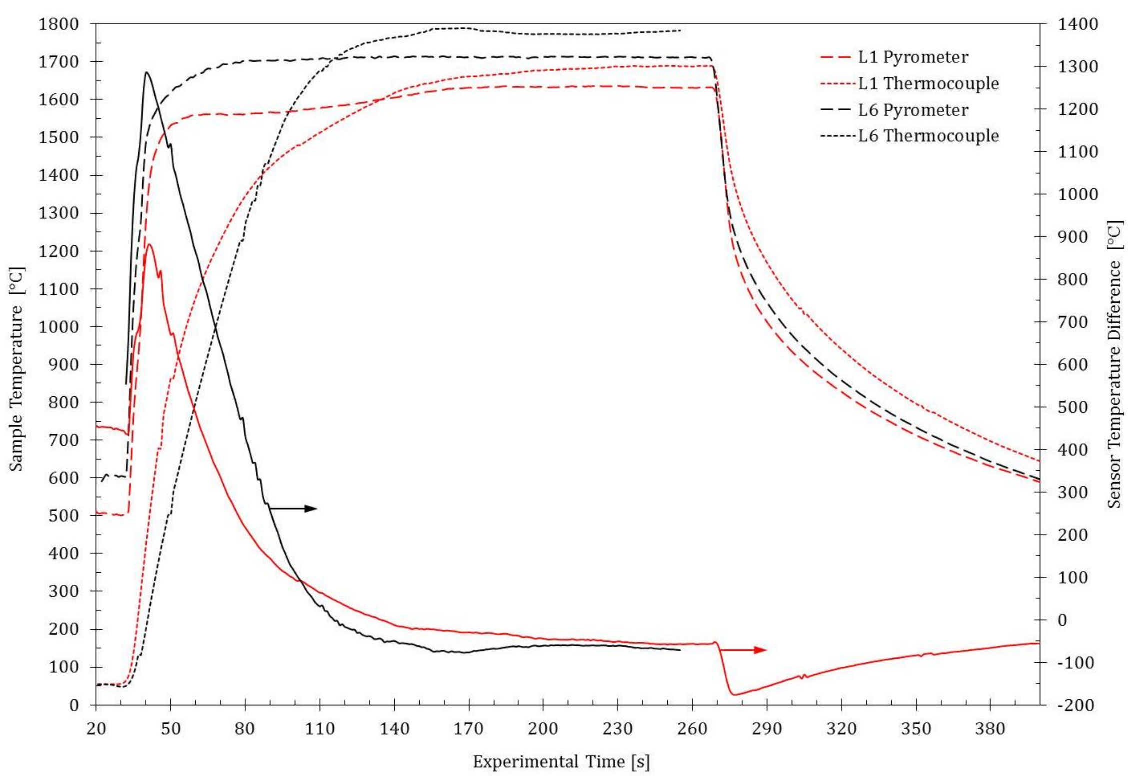

Figure 2: Dynamic surface temperature of two material samples during a solar-TG experiment. L1 material (red) is un-sintered raw ZnO powder; L6 (black) is pre-sintered ZnO powder (1300 °C for 4 hours). Surface temperature is shown as recorded by a solar-blind pyrometer (dashed lines) and type-K thermocouple (small dashed lines) on the left y-axis. Temperature difference between the pyrometer and thermocouple is shown on the right y-axis.

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