Energy Analysis of \( n \)-Dodecane Combustion in a Hetero/Homogeneous Heat-Recirculating Microreactor for Portable Power Applications

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Abstract. An energy analysis is presented for \( n \)-dodecane/air combustion in a heat recirculating Inconel microreactor under vacuum conditions. Microreactor channels are partially coated with platinum enabling operating with coupled heterogeneous and homogeneous reactions. The radiant efficiency, important for thermophotovoltaic energy conversion, was found to decrease from 57\% to 52\% over 5 different runs covering 377 min of operation. A similar decrease in combustion efficiency was observed with 6\%-8\% energy lost to incomplete combustion and 5\%-6\% lost through sensible heat in the exhaust. The remaining thermal loss is from unusable radiation and conduction through inlet and outlet tubing. Changes in the Inconel microreactor geometry and emissivity properties were observed.

1. Introduction
Hybridized thermophotovoltaic (TPV) power sources with batteries can provide lighter and longer running power systems than battery-only systems. Efficient microreactors are required to convert the high specific energy contained in tactical fuels such as JP-8 into radiated heat for portable power applications. Microreactors have challenges with efficiency at the high operating temperatures (\( >1000^\circ\text{C} \)) required for TPV conversion and stability owing to short residence times and the high radiated heat.

Hybrid hetero/homogeneous catalytic microreactors show promise as stable heat sources for portable power devices due to their ability to resist both extinction under high heat loss and blowout under high throughput conditions. Confinement and fuel decomposition were previously shown to play a critical role in determining \( n \)-dodecane combustion performance for hybrid microreactors without heat recirculation and under ambient conditions [1]. In this work we experimentally analyze the performance of a hybrid microreactor in a vacuum environment with heat recirculation to improve efficiency [2].

2. Microreactor
Planar Inconel 600 microreactors with 20 mm x 20 mm radiating surfaces designed on two sides were machined having two outer channels and one inner channel with 0.5-mm and 1.0-mm gaps respectively (figure 1). All walls are 0.5 mm thick making the total microreactor thickness 4 mm. Previous work predicted improved chemical-to-radiation efficiency for partially filled channels [3]. Thus, platinum was deposited into only 10 mm of the channels by pooling chloroplatinic acid in the microreactor for 24 hr.

The microreactors are operated in \( <10^{-5} \) Torr environment with a 100-W equivalent \( n \)-dodecane flow to mimic expected conditions in ultra-compact thermophotovoltaic sources for portable power.
applications. \( n \)-dodecane, used as a surrogate for JP-8, is injected approximately 100 mm upstream of the microreactor inlet into a dry air stream pre-heated between 423 K and 433 K. The \( n \)-dodecane/air mixture enters through the inner channel and undergoes a series of surface reactions and gas-phase decompositions once reaching the platinum coated region [1]. Heat exchanged from recirculated exhaust and conduction along the inner walls heats incoming reactants upstream and improves efficiency.

**Figure 1.** Microreactor (a) image and (b) schematic showing microreactor dimensions, fuel/air flow paths, and approximate catalyst coating location.

### 3. Energy Analysis Measurements

The microreactor energy balance in a vacuum environment includes radiation, conduction along the inlet and outlet tubes, sensible heat contained in the exhaust, and unburned products from incomplete combustion. Maximum radiant energy from the microreactor surface to a photovoltaic cell is desired for TPV conversion. The radiant efficiency, \( \eta_{\text{rad}} \), is defined as the power radiated, \( Q_{\text{rad}} \), from the 20 mm x 20 mm microreactor faces divided by the fuel input power, \( Q_{\text{chem}} \), as determined by the lower heating value. A pyrometer (Raytek MI3-2M) mounted on an X-Y stage was used to measure the surface temperature, \( T_{\text{surface}} \), through a sapphire window. The surrounding vacuum chamber wall temperature, \( T_{\text{surrounding}} \), is assumed to be significantly less than the microreactor temperature. The radiant efficiency is calculated by:

\[
\eta_{\text{rad}} = \frac{Q_{\text{rad}}}{Q_{\text{chem}}} = \varepsilon \int_{A} \left( T_{\text{surface}}^4 - T_{\text{surrounding}}^4 \right) dA.
\]

Combustion efficiency, \( \eta_{\text{comb}} \), is estimated by carbon counting of the gaseous exhaust products. The carbon-content of the liquid is assumed to be negligible. The gaseous products are analyzed using micro gas chromatography (Agilent 490) capable of measuring up to C3. The chemical-to-thermal conversion, or heat release, efficiency is estimated by

\[
\eta_{\text{heat release}} = \sum \frac{X_i}{X} n \Delta H_f(i) + n_{H_2} \Delta H_f(H_2O) - n_{C_{12}H_{26}} \Delta H_f(C_{12}H_{26}) / LHVC_{12}H_{26},
\]

where \( X/X \) is the measured molar ratio of species \( i \), \( n \) is the total molar flow rate, \( \Delta H_f \) is the heat of formation, and \( LHVC_{12}H_{26} \) is the lower heating value for \( n \)-dodecane.

The sensible heat lost in the exhaust is estimated by the temperature difference between the outlet temperature during combustion and the inlet temperature during pre-heating. K-type thermocouples were inserted into the inlet and outlet tubes to approximately 20 mm from the microreactor to measure the \( n \)-dodecane/air inlet and exhaust temperatures.

### 4. Results and Discussion

Figure 2 shows an image of the microreactor during and after sustained combustion. A bulge in the outer channel on both sides of the microreactor was observed. The bulge is caused by the stress induced by >1 atm pressure difference caused by operating in the vacuum environment and weakening of Inconel.
at high temperatures [4]. A peak deflection of 1.0 mm was measured in the central region of the microreactor suggesting the outer channel heights on both sides each increase from 0.5 mm to 1.0 mm. This change can affect the heat exchanged from the walls to and from the gas and may in turn affect the coupling between the heterogeneous and homogeneous reactions if the reactions are not completed in the inner channel [5]. Approaches to strengthen the outer microreactor walls are required to maintain the geometry integrity and may include adding in a rib structure or thickening the outer Inconel wall.

A large error exists in measuring the absolute temperature of the microreactor surface due to the unknown emissivity of Inconel 600 in our vacuum environment. The surface visually varies from an oxidized surface to a polished un-oxidized surface (figure 2b). The total emissivity can be as high as 0.82 at 1253 K [4], but data is inconclusive at high temperatures in vacuum where surface oxides can be reduced. Oxidized Inconel 718 total hemispherical emissivity measurements ranged between 0.35 and 0.5 for different oxide formation conditions at conditions up to 1200 K in vacuum [6]. Although, Inconel 718 has a lower nickel content than Inconel 600, similar oxides may form. The analysis performed in this work used an emissivity of 0.65. The actual average surface temperature can range from 1339 K to 1094 K for an emissivity of 0.4 to 0.9 respectively. The impact, however, to radiant efficiency measurements using pyrometer data is <1% for the range of emissivity since the pyrometer measures the amount of radiation absorbed by its detector.

Figure 3 shows the variation of $T_{\text{surface}}$ for an average emissivity of 0.65. A temperature drop of 103 K from the microreactor tip to the base is measured by averaging the temperature across the microreactor width. The temperature drop along the microreactor could be more significant since the surface emissivity is expected to increase from right to left causing the actual temperature to be lower towards the inlet and outlets and higher at the microreactor tip. Further studies may optimize the surface temperature and uniformity through a combination of catalyst coating location [3] within the inner and outer channels, as well as the level of confinement as determined by the channel height [1,5].

The radiant efficiency calculated from the surface temperatures in figure 3 is 57% assuming $T_{\text{surface}}$ is the same on both sides. TPV radiative to electrical conversion efficiencies of 20% and higher have been demonstrated for InGaAs and InGaAsSb photovoltaic cells [6,7] suggesting greater than 10% total conversion efficiency is feasible if combined with this microreactor. Other possible losses include cavity loss, photovoltaic cell cooling, as well as energy required to pump the fuel and air.

The radiant efficiency change over 5 different combustion runs is shown in figure 4. Each run lasted between 43 min and 100 min with the total time of 377 min. Prior to Run #1, the microreactor underwent various combustion events including propane combustion outside the vacuum chamber and ignition experiments with $n$-dodecane inside the vacuum chamber. A decrease in the combustion efficiency and heat release efficiency was observed (figure 4) suggesting a decrease in catalytic activity. Although the combustion efficiency ranged from 85% to 90% the heat release efficiency ranged from 92% to 94%. A complete evaluation of the performance versus different input power and fuel-to-air ratios is necessary to determine conditions for peak performance, expected occur with more lean conditions. Furthermore, studies comparing with a fresh microreactor operation and inspection of the catalyzed surfaces overtime is required to determine if the efficiency drop is due to catalyst deactivation, catalyst surface area reduction, or other effects.
The exhaust temperature decreased from 963 K to 923 K over the 5 runs, averaged between the two exhausts, resulting in 5.3% to 5.6% sensible heat lost in the exhaust. The observed inlet temperature was higher than the outlet temperatures for all runs. This is believed to be due to a small amount of catalyst depositing in the inlet tube during the drying process. In further experiments, not shown here, the inlet temperature reduced below the outlet suggesting the surface reactions were eliminated. The radiant efficiency did not change noticeably as a result of the possible pre-combustion in the inlet tube.

Radiation from the top and sides of the microreactor were estimated to be 15% based on the measured edge temperatures measured in figure 2. The remaining 22% is assumed to be lost by conduction along the tubes and radiation from the tubes and plenum surfaces.

5. Conclusion

The performance of a microreactor combining heat recirculation and hybrid hetero/homogeneous reactions via channels partially coated with platinum was evaluated for n-dodecane fuel and stoichiometric air flow. The Inconel microreactor requires strengthening to maintain geometrical integrity for vacuum operation. Radiant efficiency up to 57% with a 94% heat release efficiency has been demonstrated. Both the radiant efficiency and the combustion efficiency were observed to reduce over time suggesting reduced catalytic activity, but further experiments are necessary to determine the cause of the degradation. The sensible heat lost in the exhaust was measured to be 5%, and the remaining 32% was lost to unusable radiation and through conduction along the inlet and outlet tubing.

6. References

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