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Raman Spectroscopy for the On-Line Analysis of Oxidation States of Oxygen Carrier Particles

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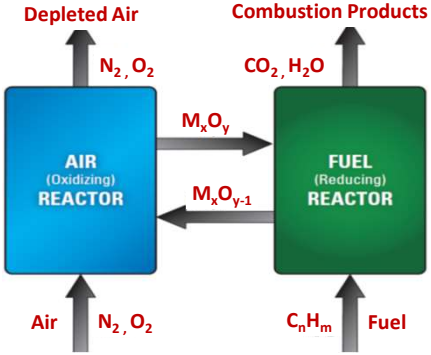
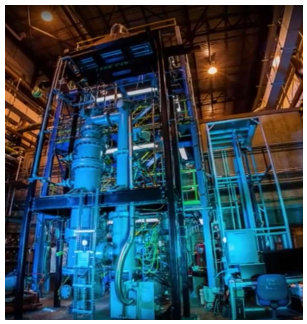
Introduction

- Need for cleaner energy systems is driving the current development of chemical looping combustion (CLC) systems.
- CLC systems use oxygen carrier particles (OCPs).



"DOE/NETL Advanced Combustion Systems: Chemical Looping Summary," July 2013, DOE/NETL



NETL Chemical Looping Reactor
https://www.youtube.com/watch?v=1r1VrtKB-AM, accessed 9/20/16

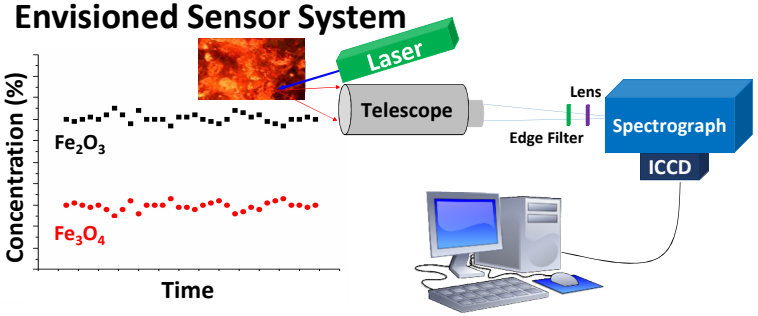
Research Challenges

- Process optimization requires knowledge of OCP oxidation state at different stages of CLC process.
- Ability to make on-line measurements of oxidation states of OCPs is lacking and new sensors need to be developed.
- OCPs are at extreme conditions, including temperatures as high as 1000 °C and pressures of 10 atm.

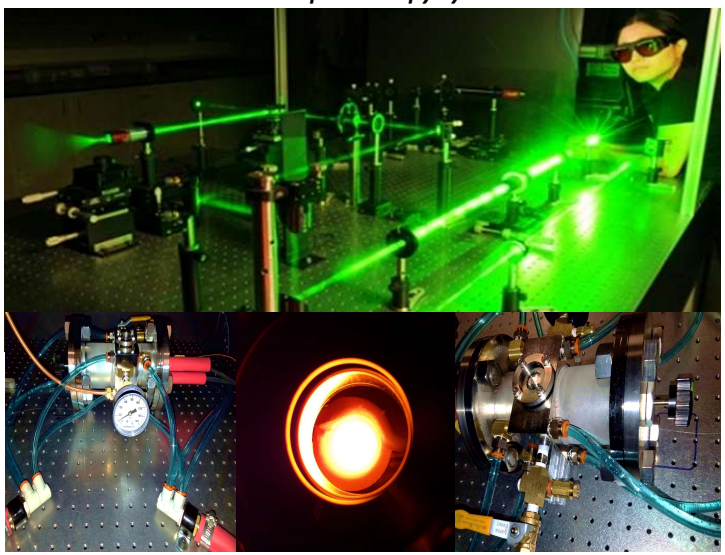
Objectives

- Set up a pulsed time-gated Raman spectroscopy system in combination with a pressurized high-temperature sample chamber.
- Optimize Raman spectroscopy system and measure high-temperature Raman spectra of OCPs.
- Develop an analysis procedure to interpret the Raman spectra.

Envisioned Sensor System

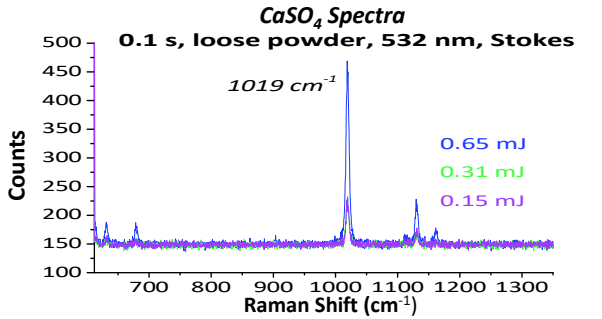


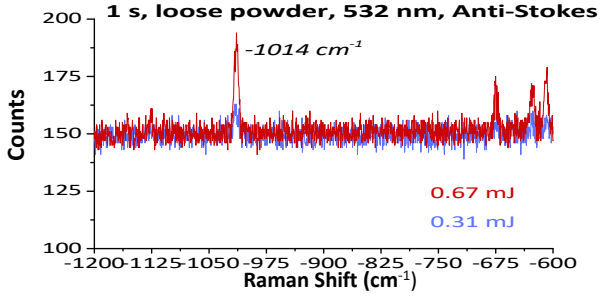
Raman Spectroscopy System

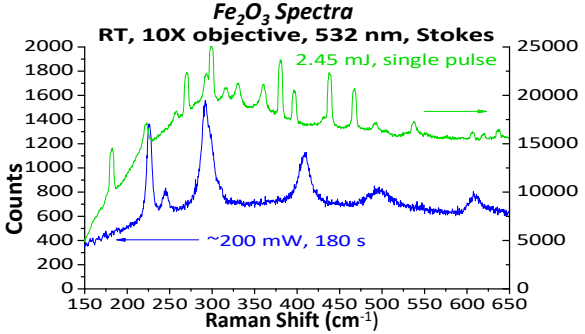


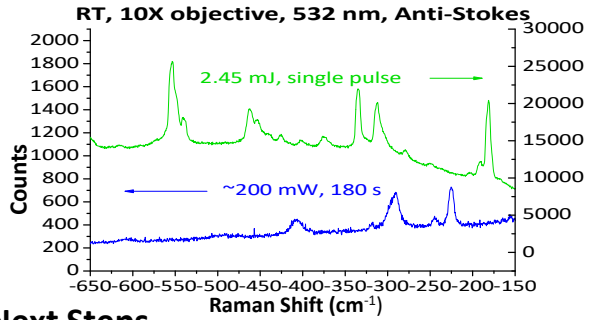
Front-view | Looking into window | Top-view

Preliminary Testing









Next Steps

- Evaluate and integrate best approach for collecting single-shot Raman spectra.
- Heat known materials (e.g. Fe_2O_3, Fe_3O_4) to high temperatures (e.g. 800°C, 900°C, 1000°C) and measure Raman spectra.
- Perform inverse calibration to determine composition and temperature.
- Conduct blind tests to demonstrate and evaluate the performance of our approach for identifying the oxidation state of OCPs at high temperature.

SUCCESS CRITERIA AT DECISION POINTS

• **End of month 12:**	Experimental Raman spectroscopy system for high-temperature measurements operational.
• **End of month 21:**	Automated analysis process operational.
• **End of month 24:**	Demonstrated high-temperature Raman spectroscopy system on a minimum of three blind samples.

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