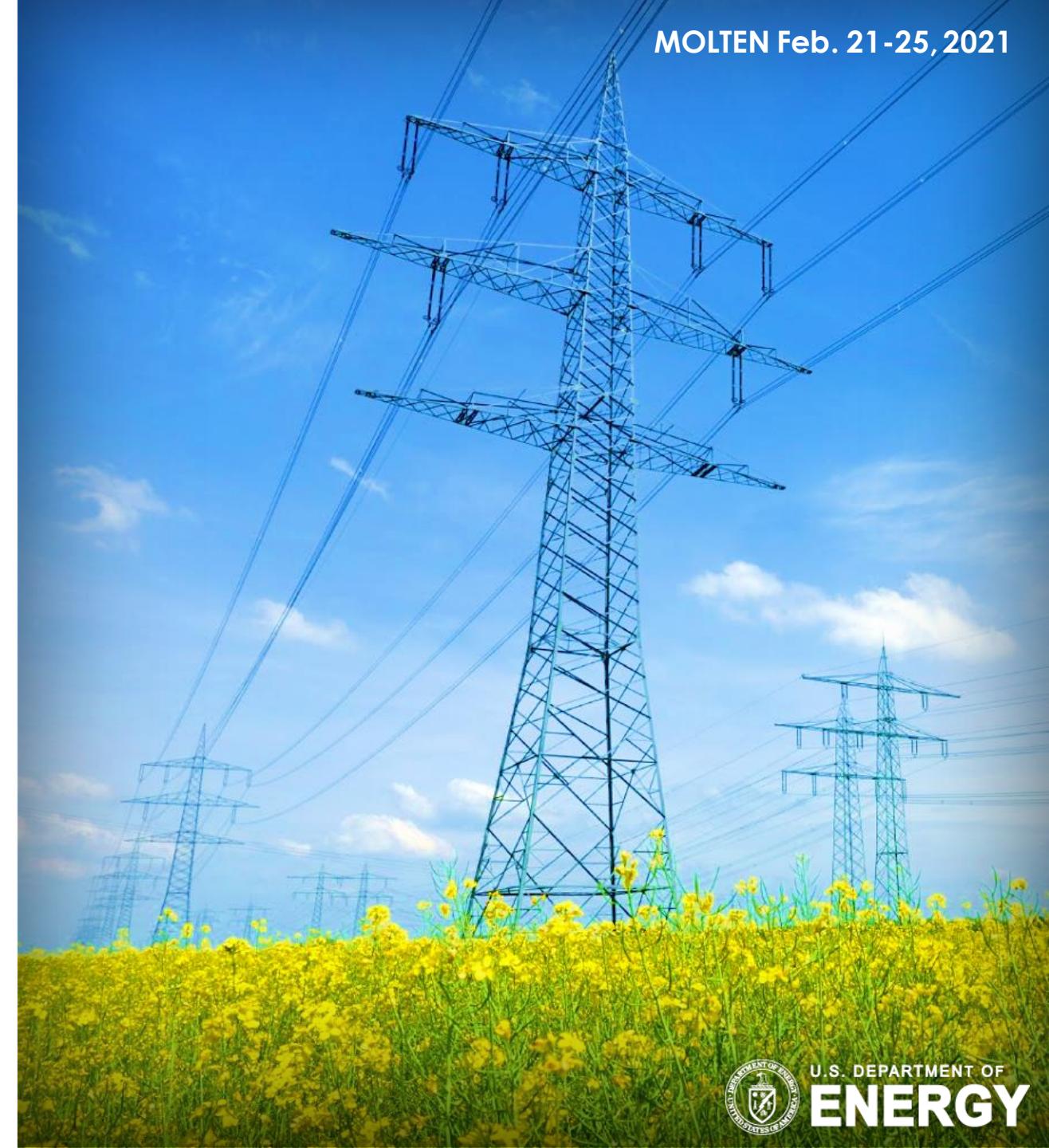


Real time evolutions of individual industrial coal particles in varied oxygen partial pressure environments

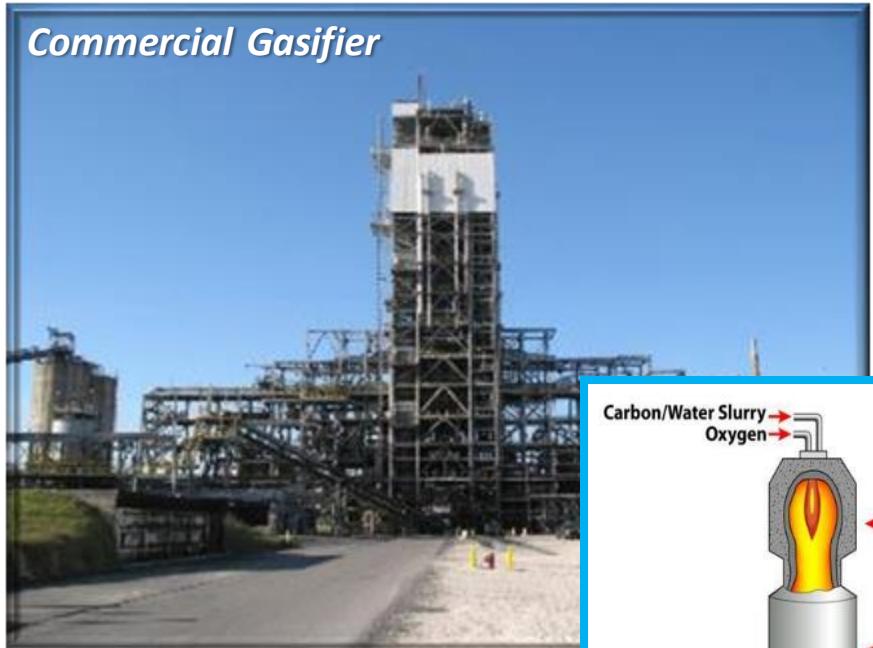
Anna Nakano^{1,2}, Jinichiro Nakano^{1,2}, James Bennett¹

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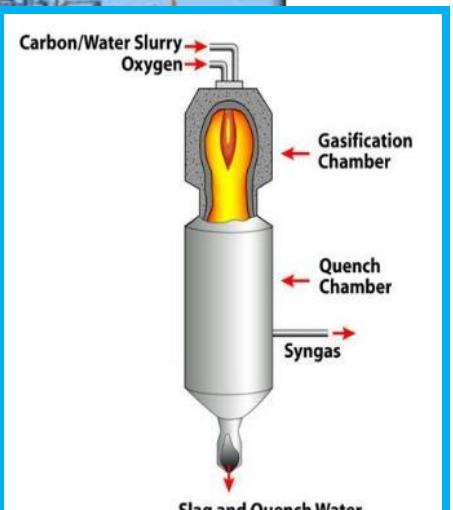


Introduction

- Example of commercial use of coal

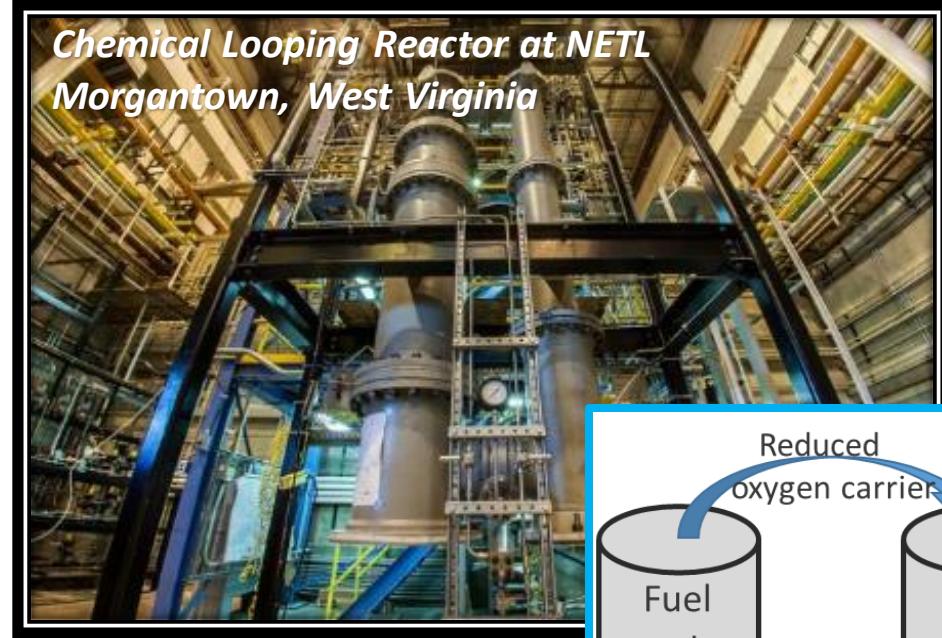


Gasification is used to convert a carbon feedstock into the primary product of CO and H₂ for power and chemical generation.

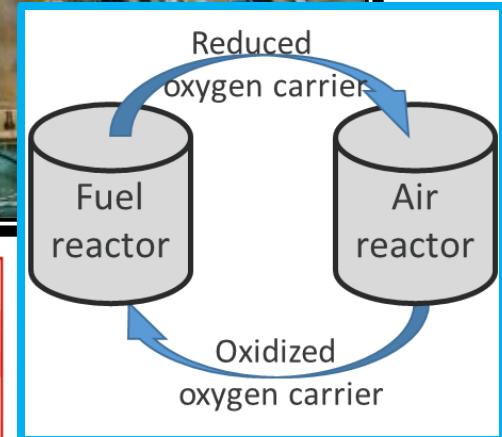


Some Current Gasifier Design

- Alternative use of coal



Chemical looping combustion (CLC) is an alternative concept for energy generation. Fuel: reducing gas or solid carbon feedstock



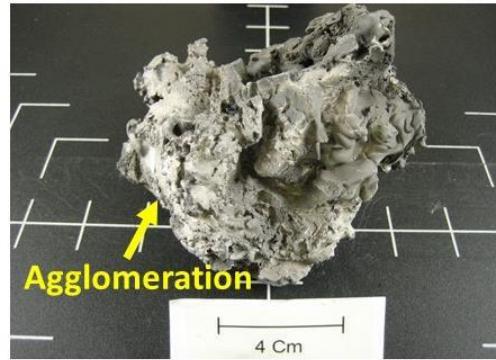
Introduction

Motivation

- On heating, volatiles and other impurities (ash) evolve from the coal structure promoting a number of issues that include fouling, agglomeration and slag.
- A better understanding of coal evolution behavior would help improve current gasification systems and enable new systems such as chemical looping combustion (CLC).



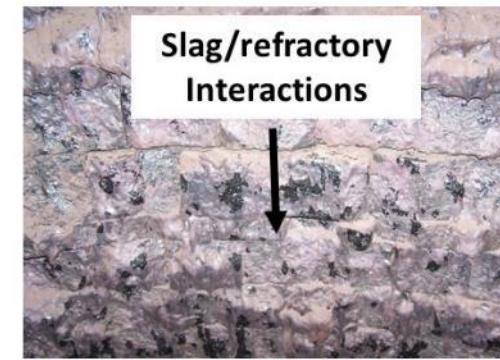
Heat Exchanger



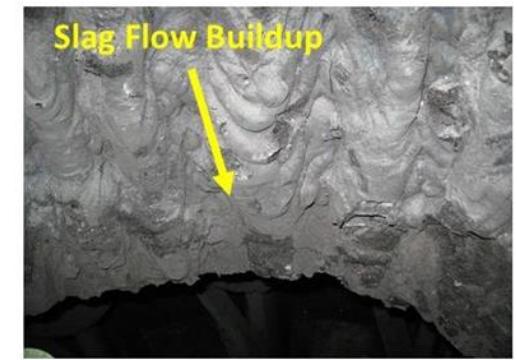
Fluidized Bed

Objectives of this work

- Visual investigations of the thermal behavior of commercial bituminous coal as a potential CLC feedstock under varied oxygen partial pressure environments by using the HT-CSLM capabilities.
- The fundamental data collected in this work is to guide the potential use of coal as carbon feedstock for the CLC process.



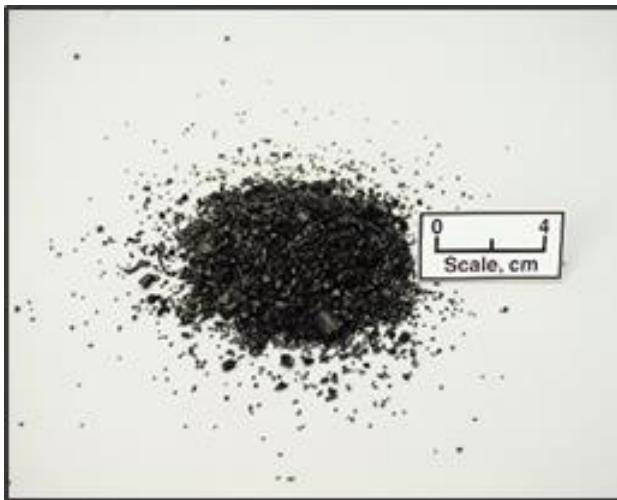
Gasifier Sidewall



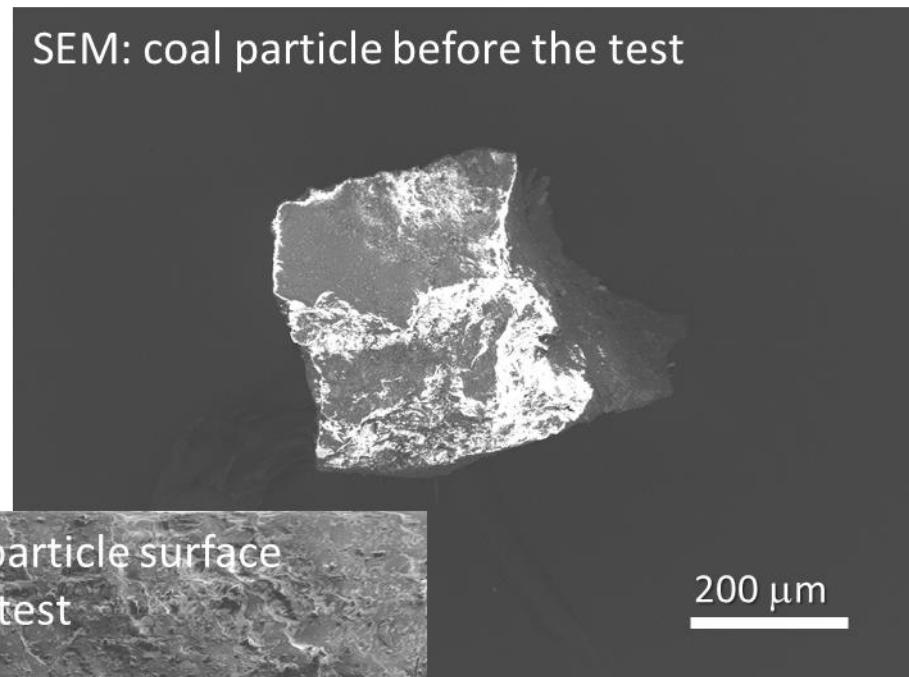
Gasifier Sidewall

Experimental. Materials

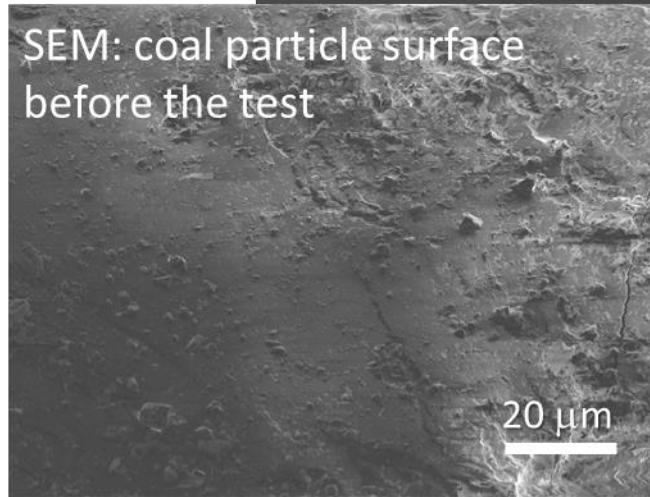
Ground commercial bituminous coal
individual particles was collected
from a gasifier in the US.



SEM: coal particle before the test



SEM: coal particle surface
before the test



Medium-volatile bituminous coal*



Proximate and ultimate analyses (mass%)

	Arithmetic Mean	Minimum	Maximum
Moisture	2.7	0.9	6.2
Volatile matter	24.0	20.8	27.8
Fixed carbon	66.8	59.9	72.3
Ash	6.4	3.6	11.4
Hydrogen	4.8	4.5	5.1
Carbon	80.9	70.8	85.3
Nitrogen	1.2	1.0	1.4
Oxygen	5.9	3.9	11.7
Total sulfur	0.7	0.5	1.1

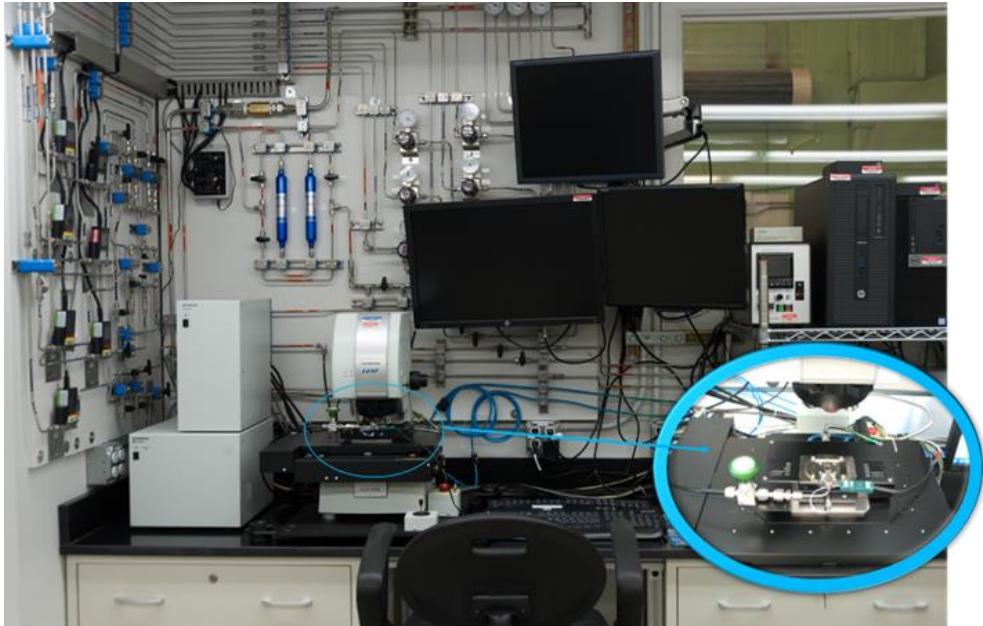
Ash chemistry (mass%)

Oxide	Arithmetic Mean
SiO ₂	41.20
Al ₂ O ₃	26.80
CaO	3.57
MgO	1.47
Na ₂ O	0.56
K ₂ O	1.07
Fe ₂ O ₃	10.4
MnO	0.02
TiO ₂	1.40
SO ₃	2.70

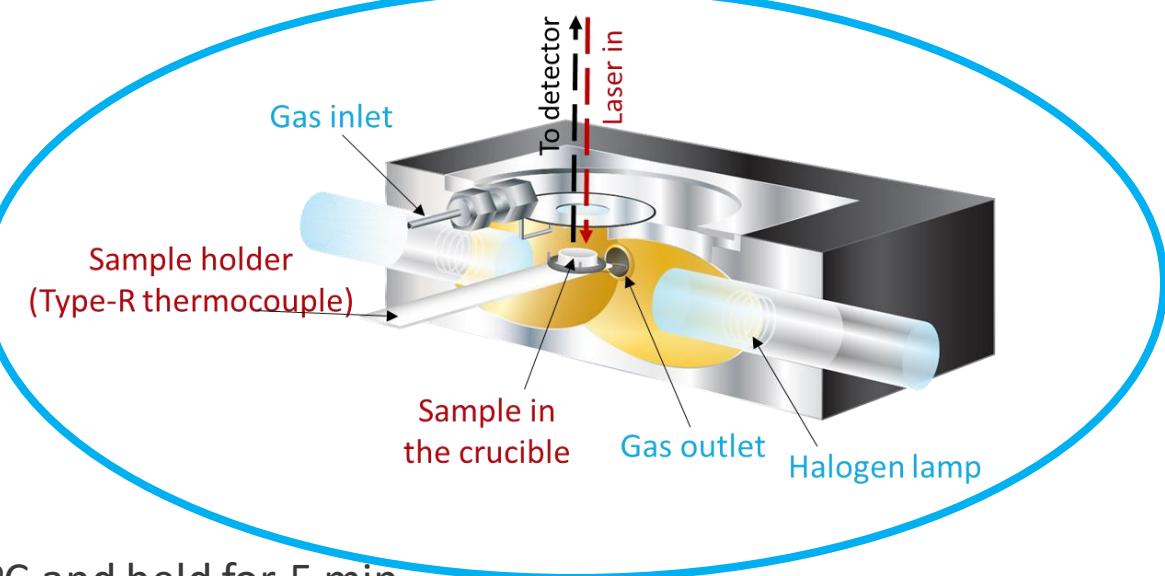
*Chemical Analyses and Physical Properties of 12 Coal Samples from the Pocahontas Field, Tazewell County, Virginia, and McDowell County, West Virginia, GEOLOGICAL SURVEY BULLETIN 1528, UNITED STATES GOVERNMENT PRINTING OFFICE, WASHINGTON : 1982

Experimental Test Procedures

HT Confocal Scanning Laser Microscope.



Heating chamber



- Individual coal particles heated at 800 °C/min to about 800 °C and held for 5 min.
- Samples quenched at different temperature on heating and after the hold.
- Gas environment: air, 82 at.% CO/18 at.% CO₂.
- Gas flow rate: 50 ml/min.
- The evolved liquid matters analyzed from the imprint on the substrate.

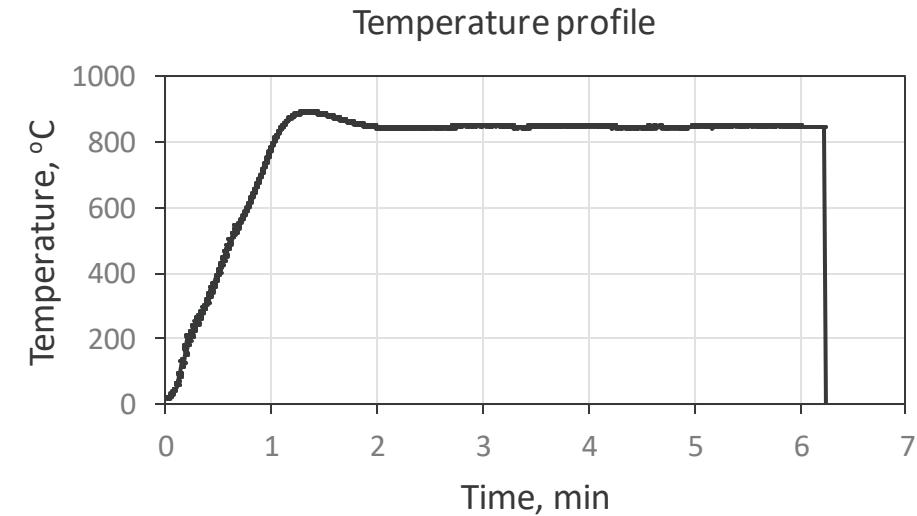
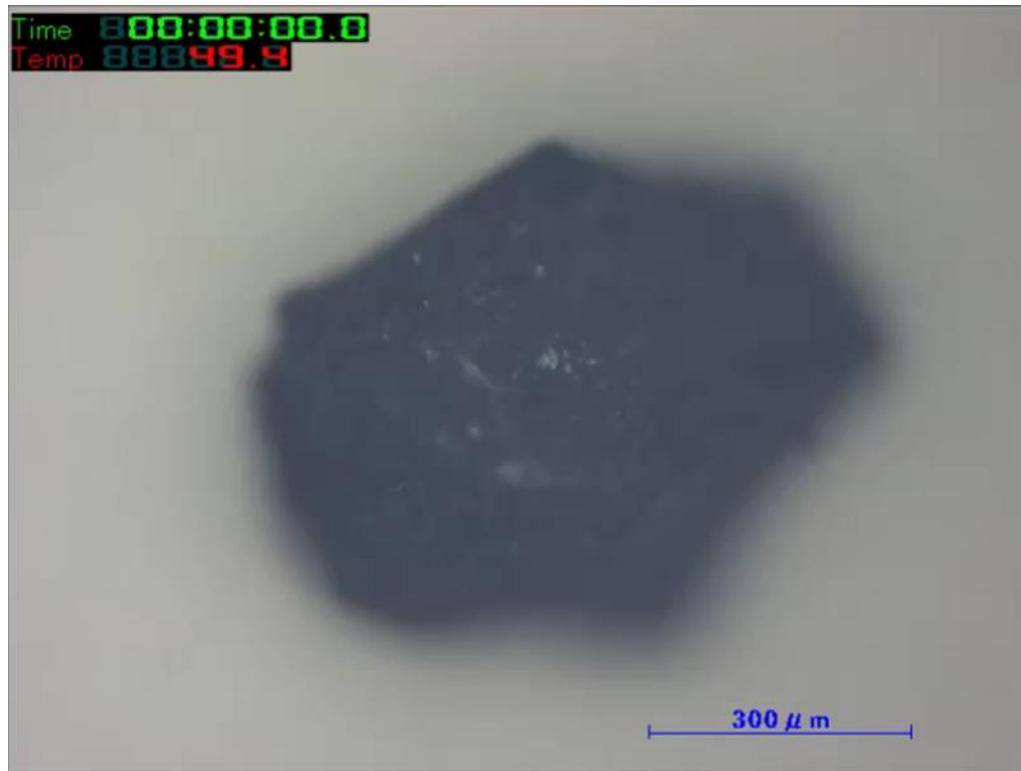
In-Situ Microscopic Study. Air



Individual coal particle behavior on heating in oxidizing environment.

white light movie

Movie speed: 4x

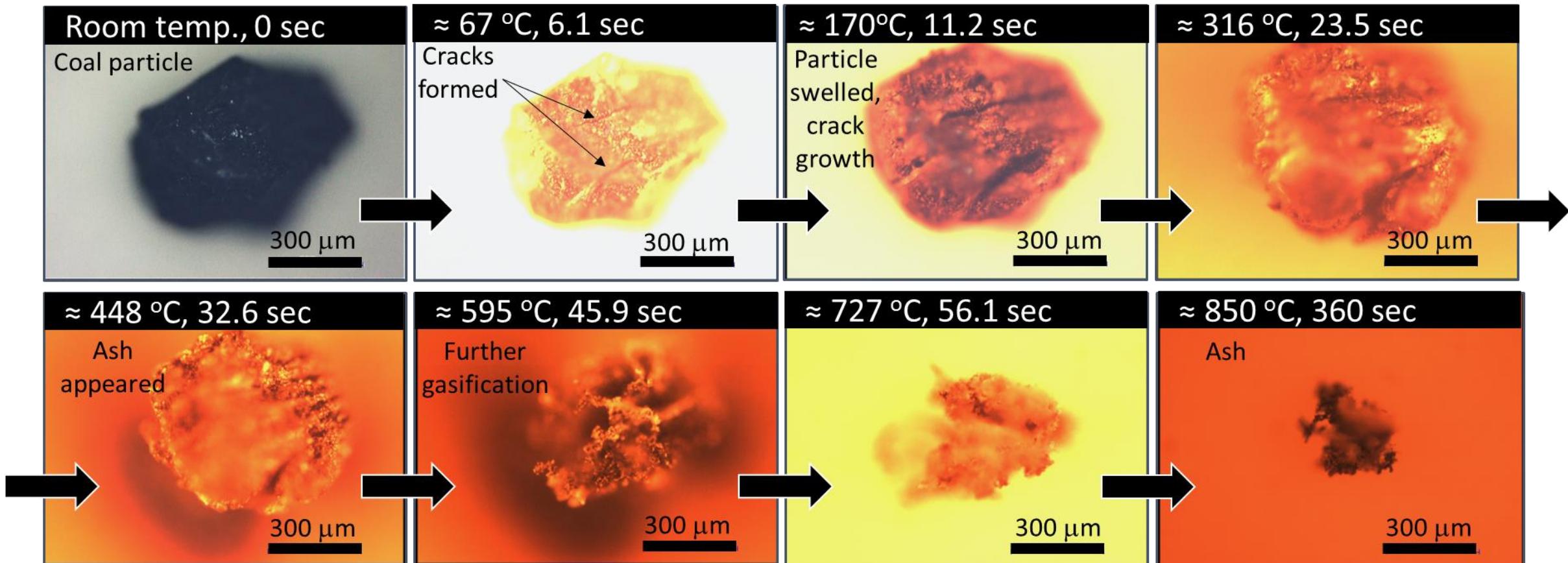


Note: bright light flashing during the first part of heating is due to the pulsive current application designed by the software to minimize overheating at low temperatures under fast heating rates.

In-Situ Microscopic Study. Air

Individual coal particle behavior on heating in oxidizing environment.

white light images



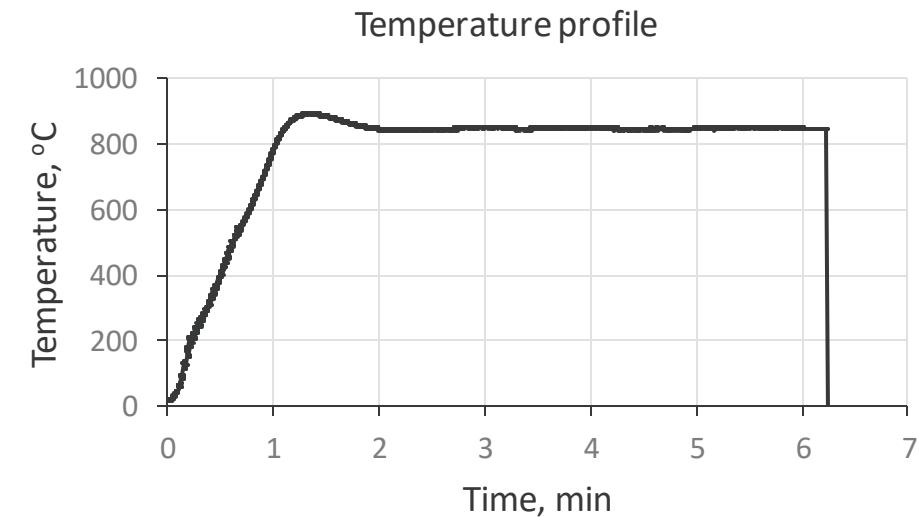
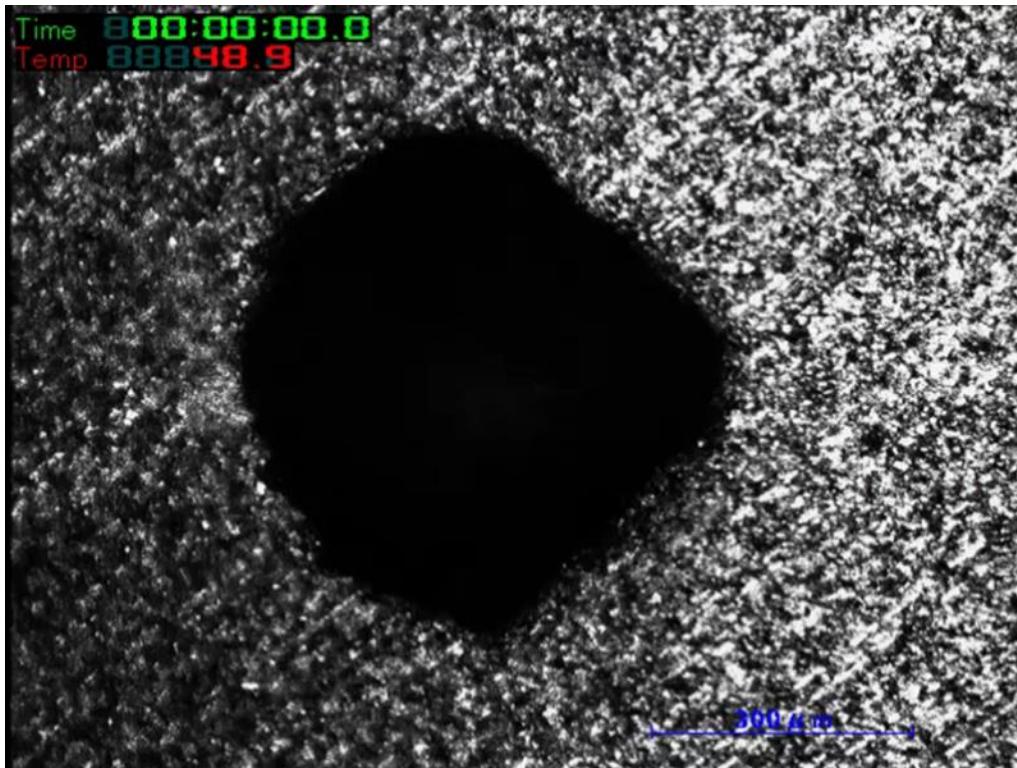
In-Situ Microscopic Study. Air



Individual coal particle behavior on heating in oxidizing environment.

Confocal **laser** movie

Movie speed:1x

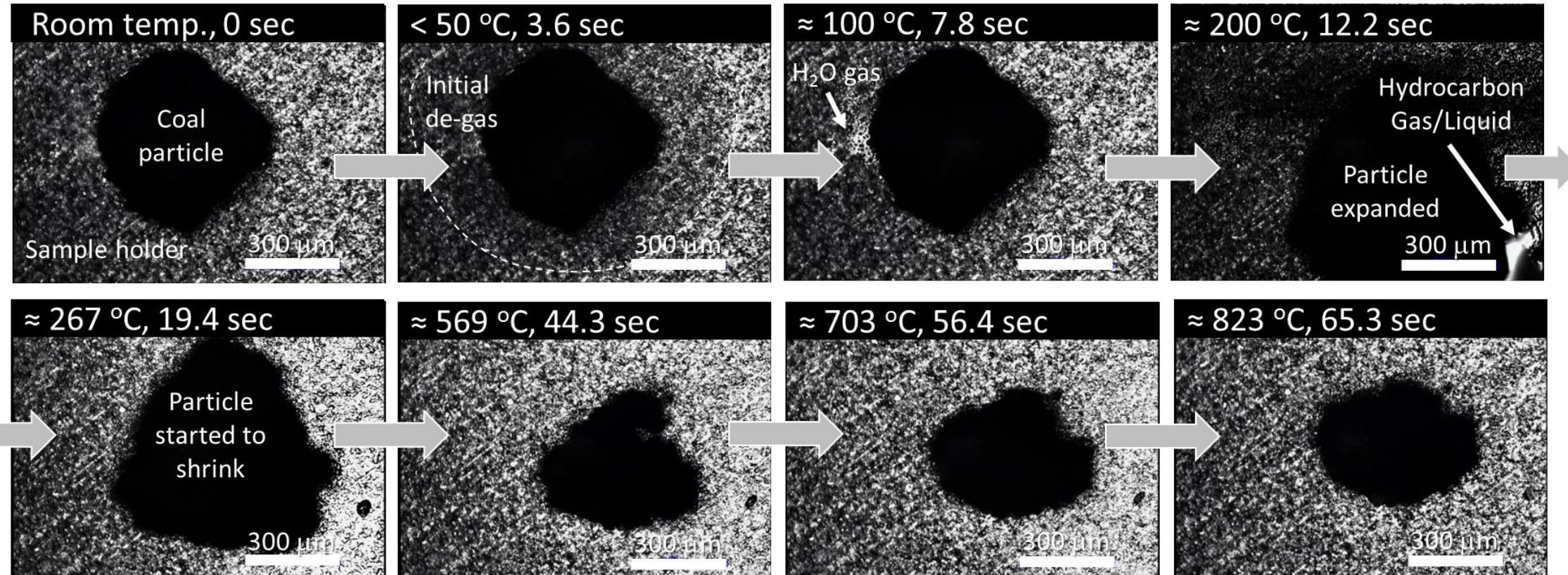


Note: focus is at the alumina spacer allows to see particle outline as a black shade along with degassed vapors.

In-Situ Microscopic Study. Air

Individual coal particle behavior on heating in oxidizing environment.

Confocal **laser** images

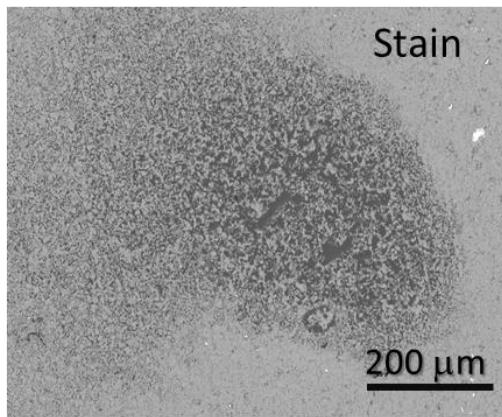


Laser (low wavelength) enabled visualization of volatile evolution and de-gassing.

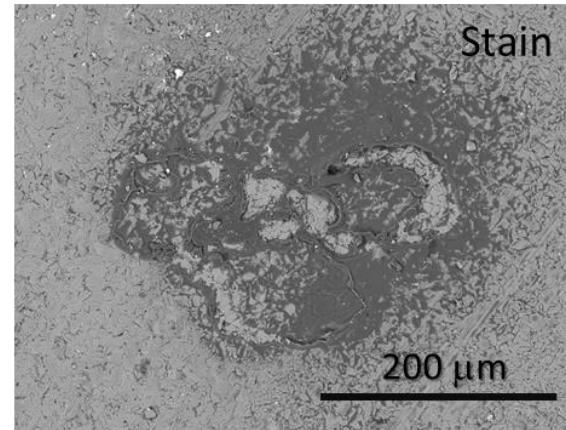
Post analysis: imprint on the substrate

SEM-EDX

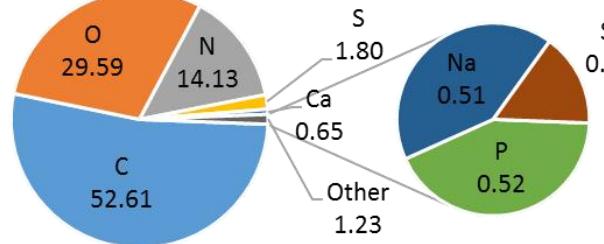
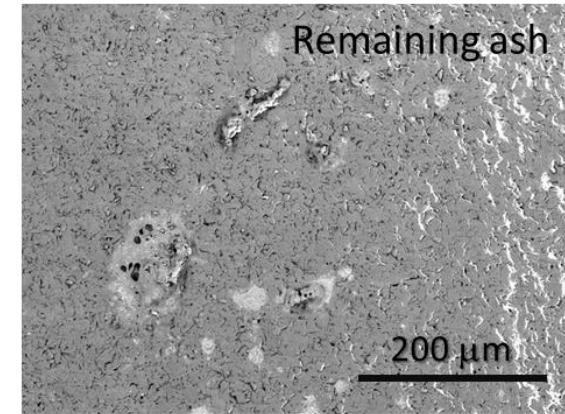
≈ 270 °C



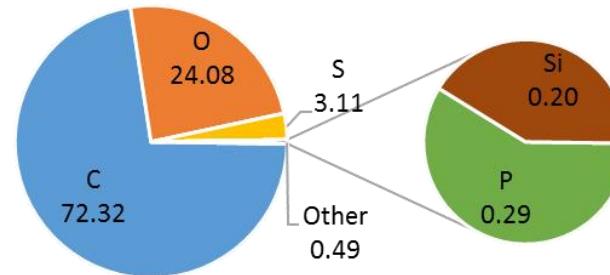
≈ 470 °C



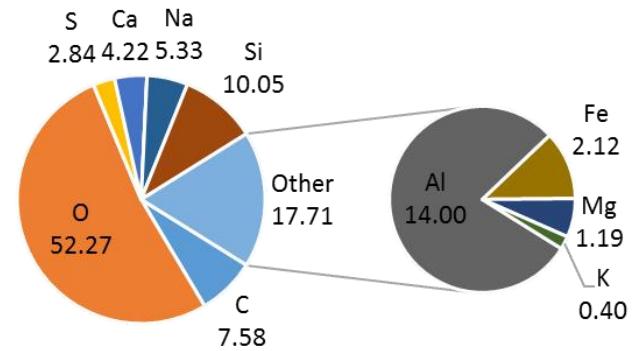
≈ 850 °C



Averaged elemental distribution (mass %)



Averaged elemental distribution (mass %)

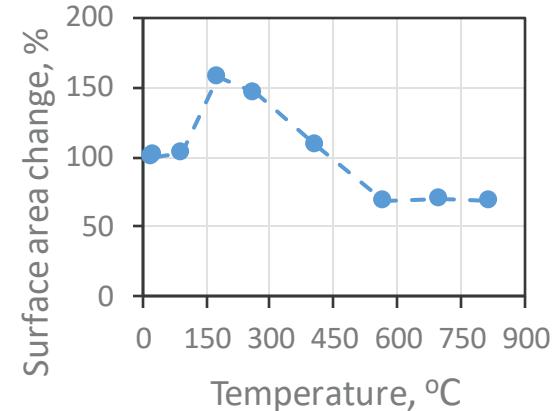
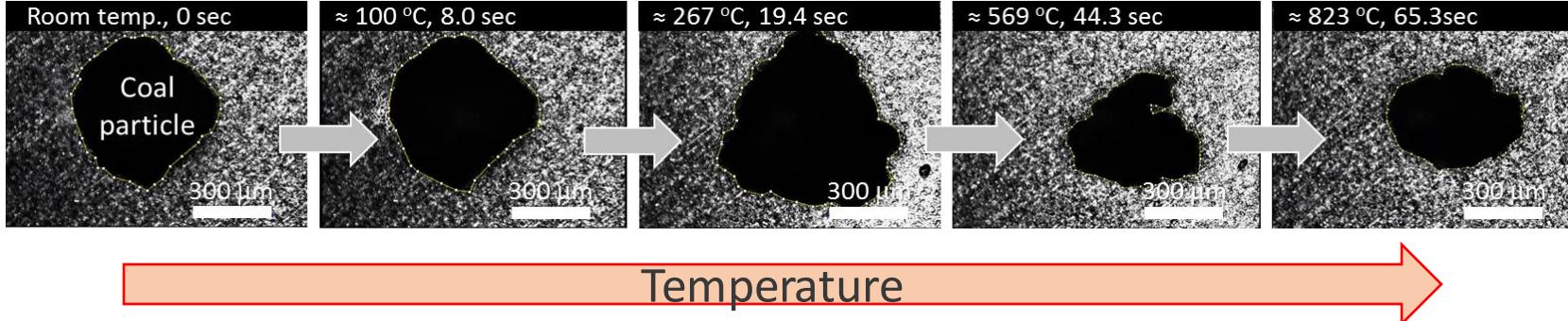


Averaged elemental distribution (mass %)

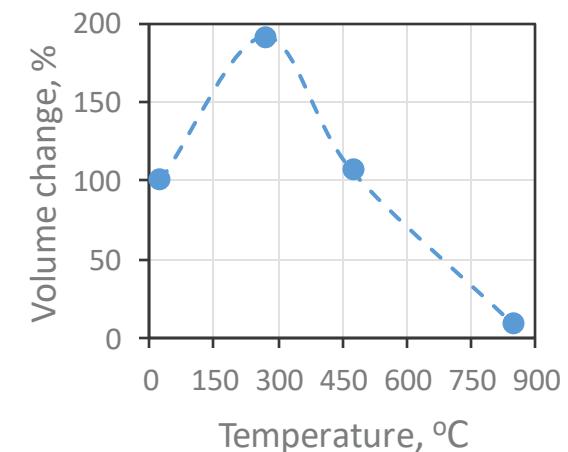
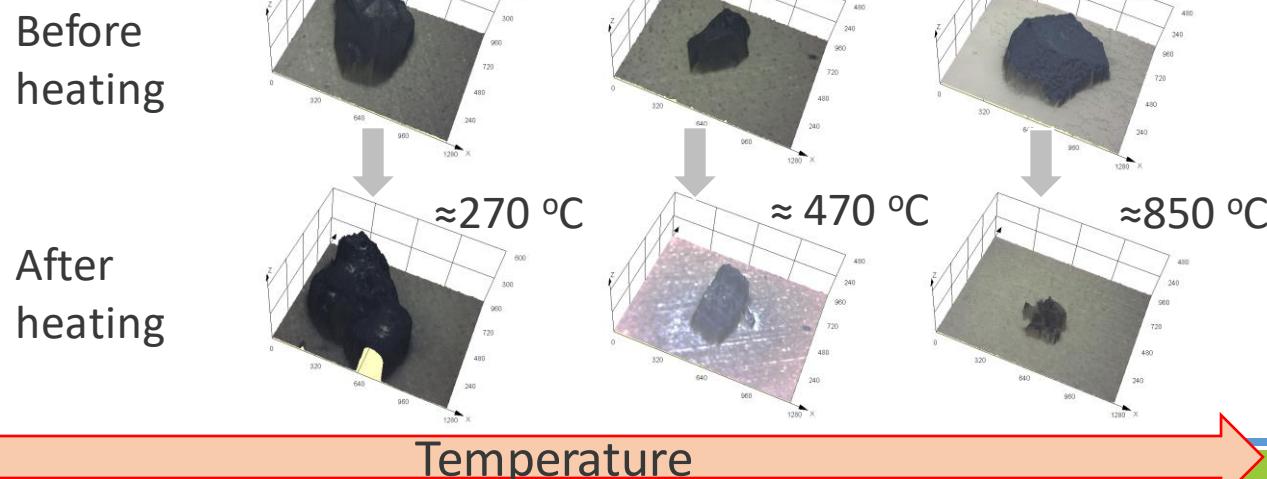
* Average of point analysis (at least 4 points) on the stains or ash.

Change in area/volume with temperature. Air

Area change with temperature. 2D confocal laser images.



Volume change with temperature. 3D laser/white light scans.



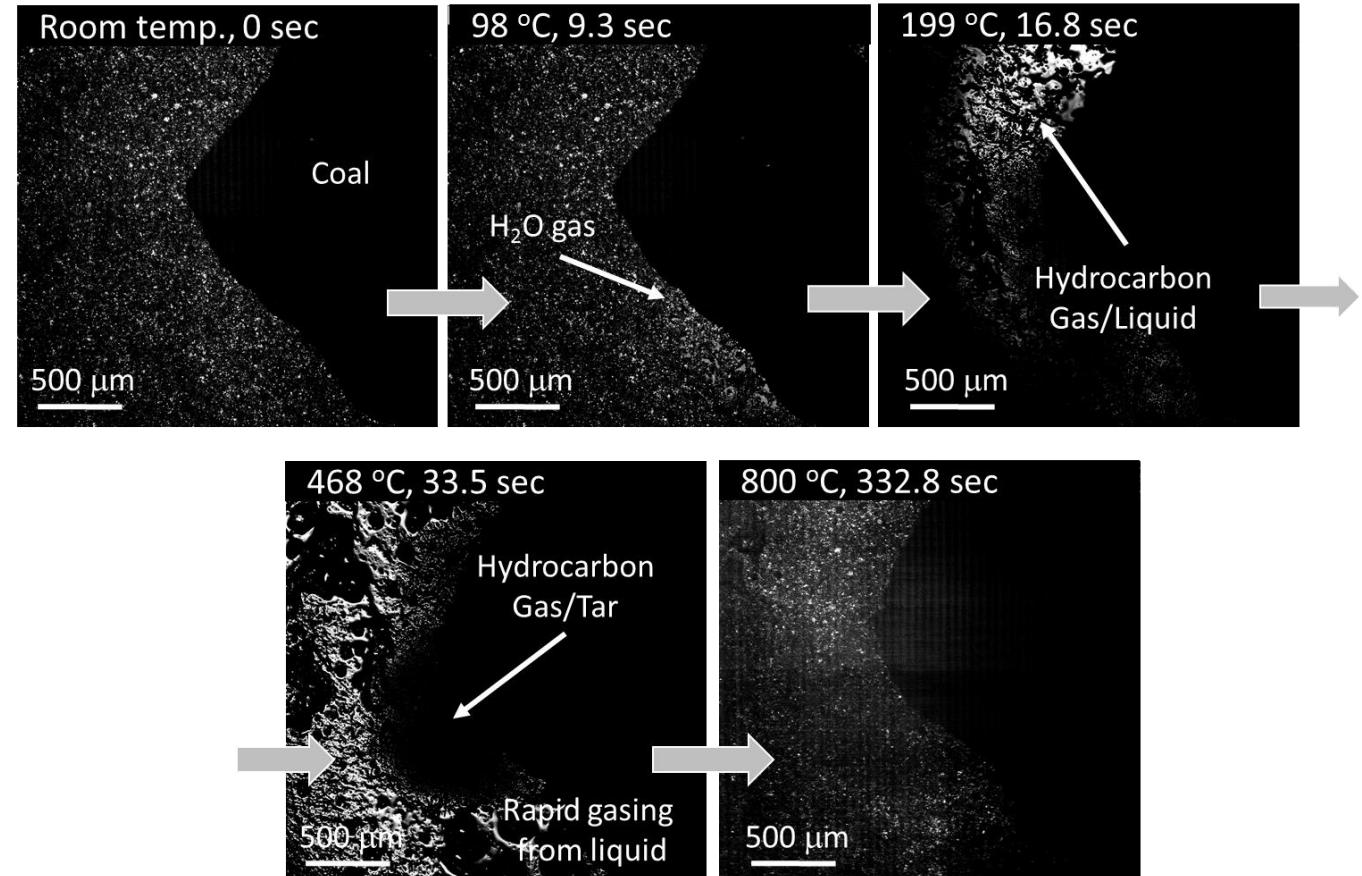
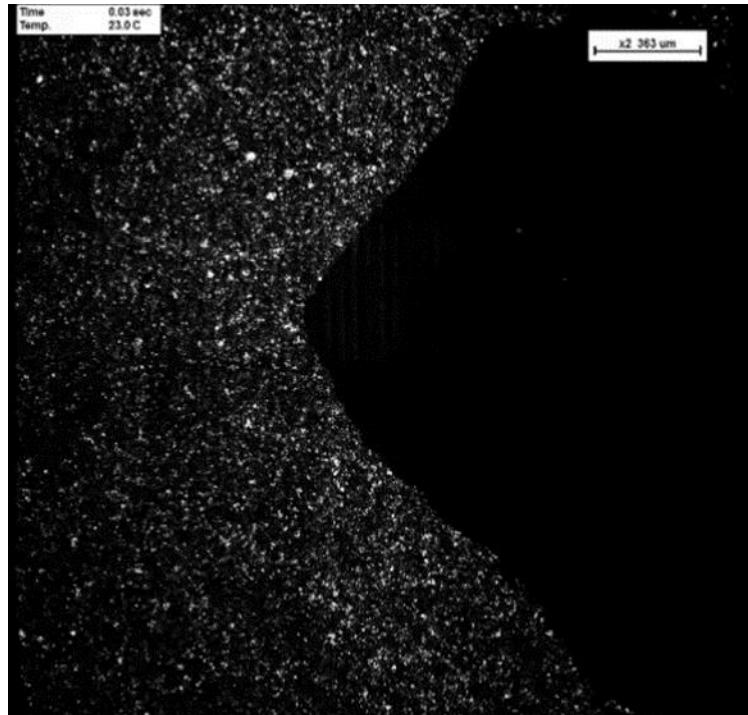
In-Situ Microscopic Study. 82 at.% CO/CO₂



Individual coal particle behavior on heating in reducing environment.

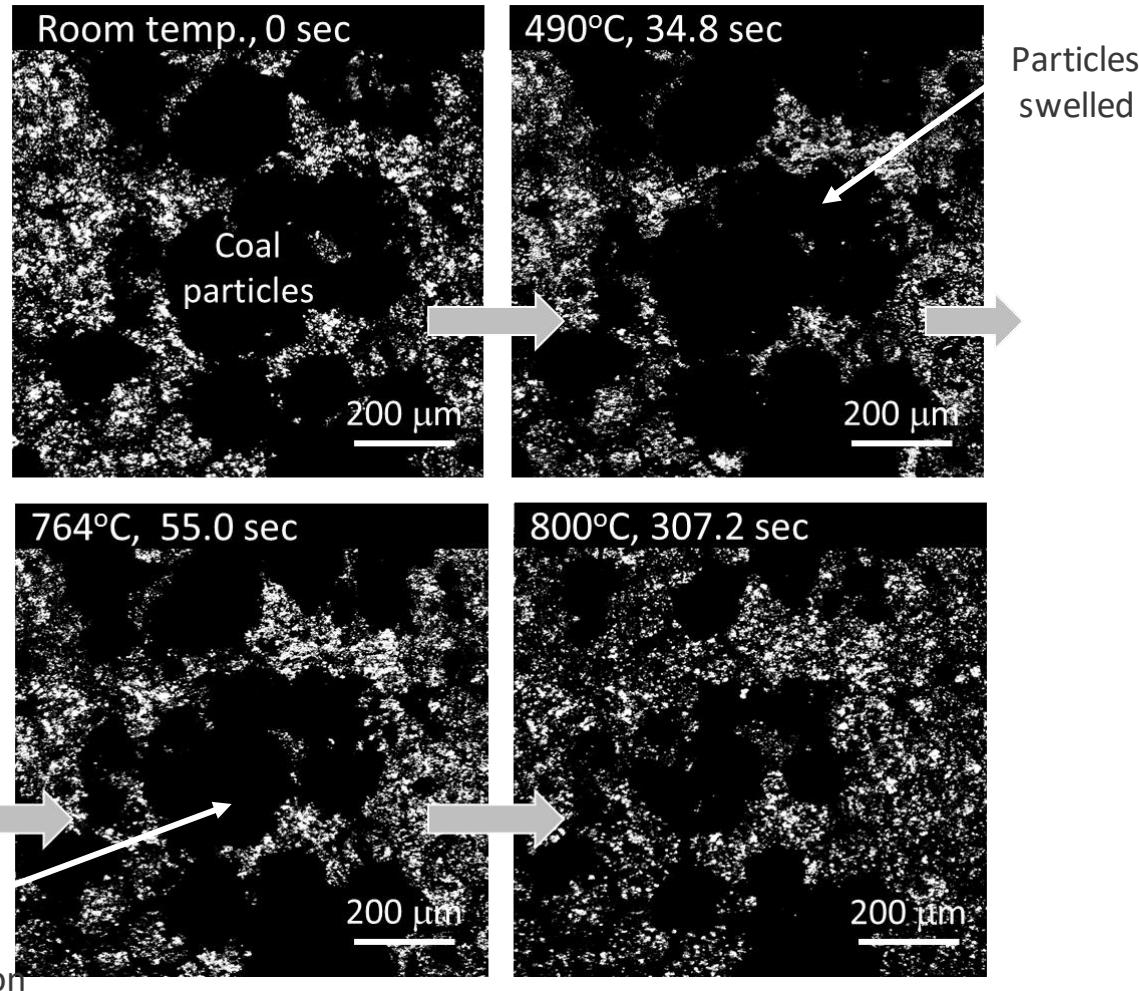
Confocal *laser*

Movie speed:16x

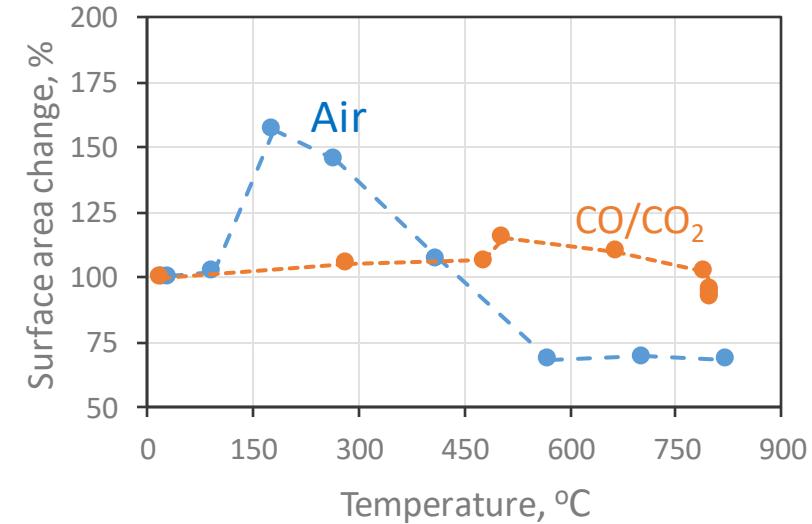


Change in surface area/volume with temperature

82 at.% CO/CO₂



Coal particle expansion/contraction



Thermal breakdown of bituminous coal (this work)



Air

- < 50°C – absorbed water removal.
- ≈ 100°C – drying continues; some particles experienced cracking.
- > ≈ 200°C – hydrocarbon gas/liquid (N-, Na-, P-, S-, Si-compounds) formation, which oxidize rapidly in the gaseous phase; particles began to swell.
- ≈ 200-300°C – observed peak of particle surface area expansion.
- > 300-400°C – particle began to shrink; ash appeared.
- > 700°C – particle shrinking slowed down.
- > 800°C – evolved liquid volatilized, leaving a carbon residue and ash behind.

82 at.% CO/CO₂

- ≈ 100°C – absorbed water removal.
- > ≈ 200°C – hydrocarbon gas/liquid formation; particles began to swell.
- ≈ 480-500°C – observed peak of particle surface area expansion; some particles experienced cracking.
- > 500°C – particle started to shrink; evolved liquids volatilized, leaving a carbon residue and ash behind.
- ≈ 800°C – particle continued to shrink.

Conclusions



- Individual bituminous coal particles were dynamically analyzed *in situ* under air and reducing CO-CO₂ gas mixture using the environmental confocal scanning laser microscope equipped with the custom made heating stage.
- Combination of the color and laser video recording enabled real-time observation of coal particle's morphological changes involving ash formation and gas/moisture/liquid evolutions as a results of heating in different oxygen partial pressure environments, which was not possible without laser.
- The evolved liquid matters were analyzed from the imprint on the substrate using SEM-EDS which provided temporal and special insights on qualitative and quantitative analysis of the coal particle thermal breakdown.
- Based on present finding and literature data, the thermal breakdown path of bituminous coal in air and CO-CO₂ gas was suggested.
- The fundamental data collected in this work would help improve existing gasification systems and enable the potential use of coal as carbon feedstock for the CLC process.

Acknowledgement

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The Authors acknowledge Mr. K. Collins (NETL) for SEM-WDS and Mr. M. Fortner (NETL) for metallography.

Disclaimer

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