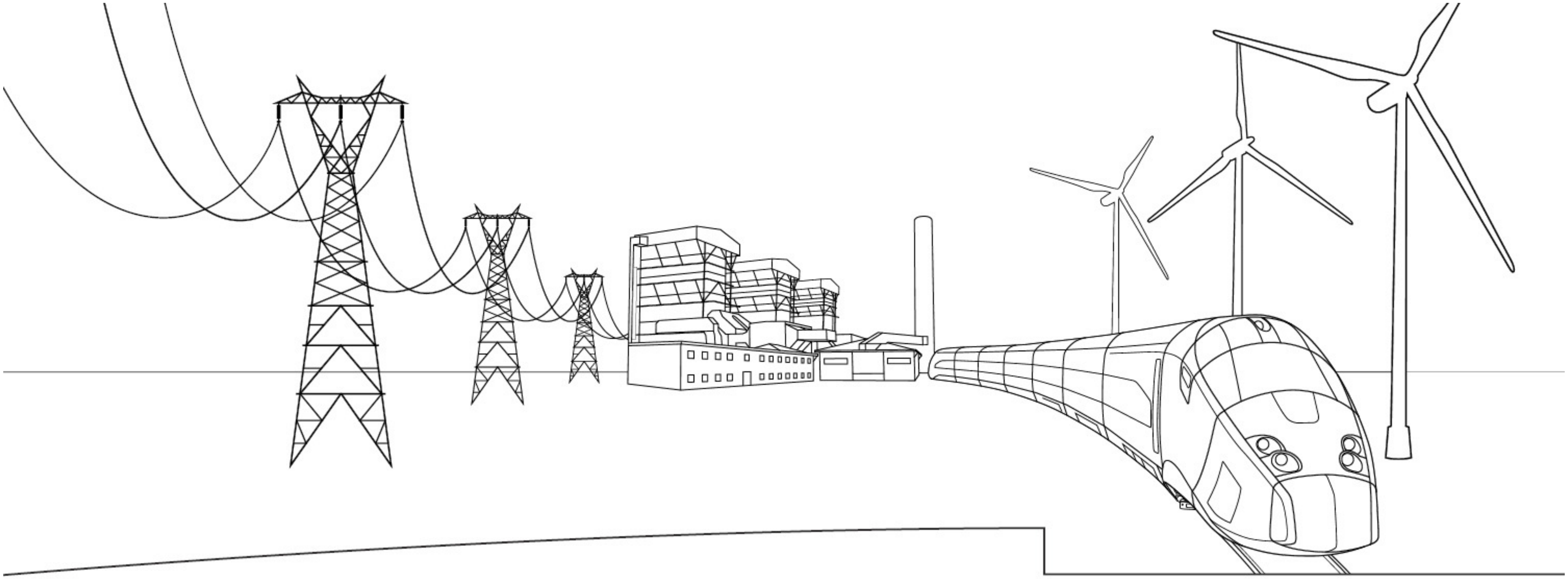

IMPROVEMENT OF ALSTOM'S LIMESTONE-BASED CHEMICAL LOOPING COMBUSTION PROCESS FOR HIGHER PURITY FLUE GAS PRODUCTION (DE-FE0025073)

Frederic Vitse

US DOE/NETL Webex Kick-off meeting

22 Oct 2015

ALSTOM
Shaping the future

AGENDA

- Background
  - What's Chemical Looping?
  - Why Chemical Looping?
  - Alstom's Chemical Looping Roadmap
- Technical approach to improved Gas Purity
- Project objective
- Project structure
- Project schedule
- Project budget
- Project Management Plan including Risk Management

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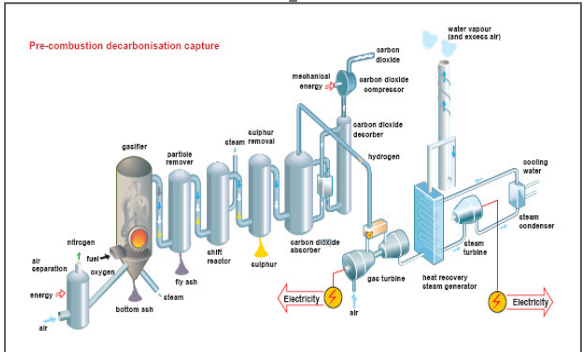
Carbon Capture Solutions

Zero emission technology pathways – Three (3) generic

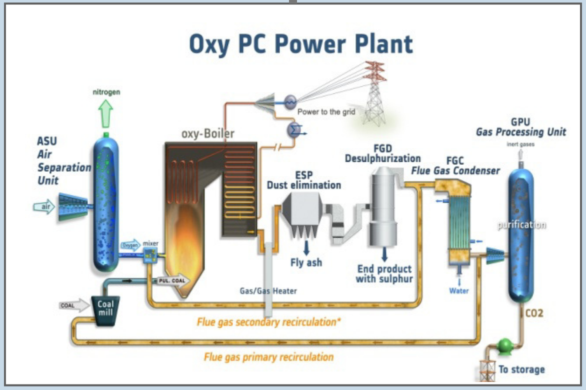
Power Plant with CO_2 Capture

Pre-combustion

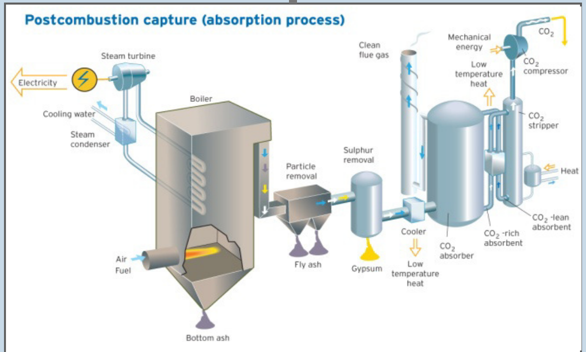


Source: Vattenfall

Oxy-combustion (New + retrofit)



Post-combustion (New + retrofit)



Source: Vattenfall

Alstom development:

- Oxy Combustion
- Chemical Looping
- Advanced Amine
- Chilled Ammonia

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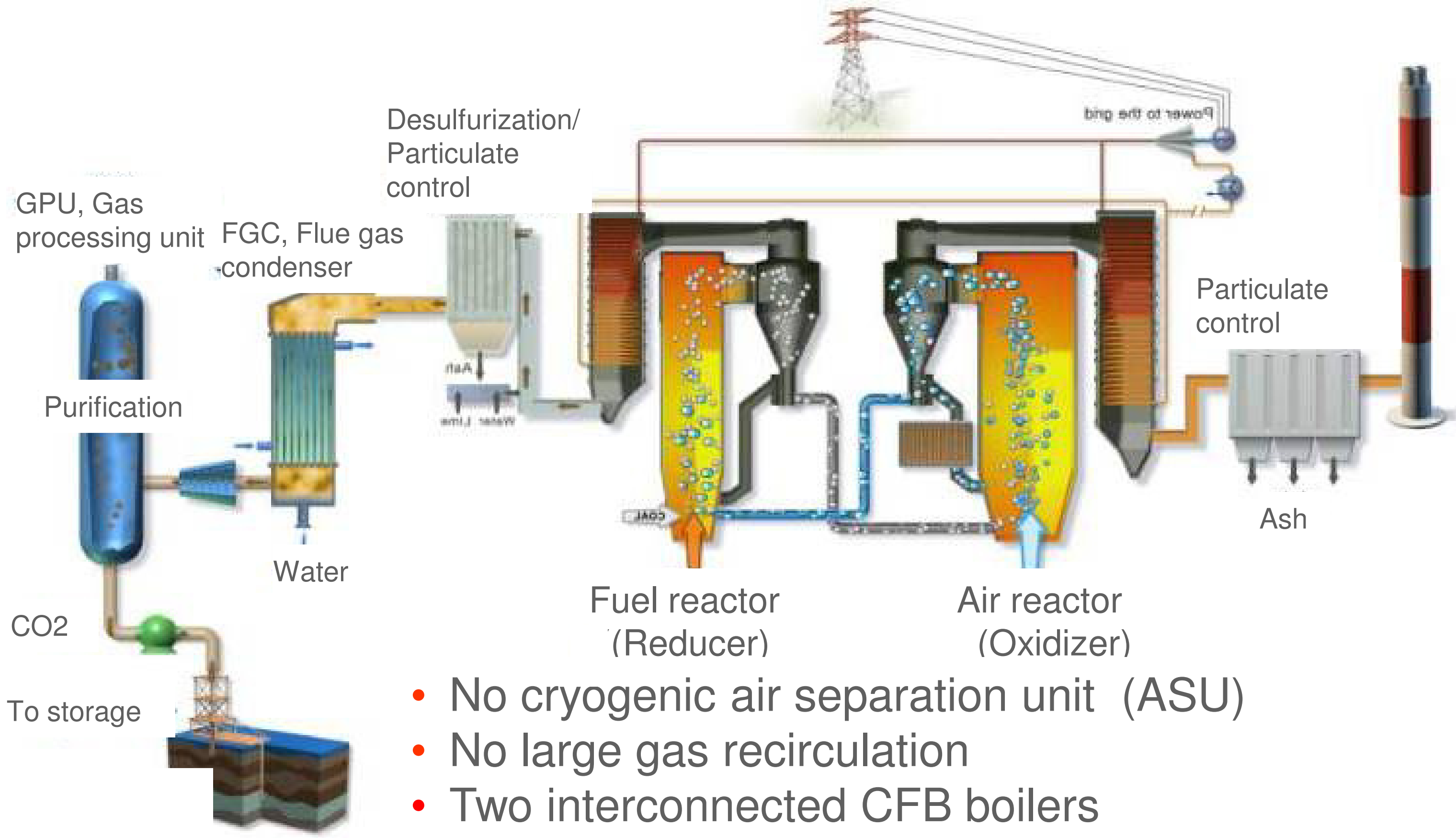
Chemical Looping Process
Advanced oxy combustion technology without ASUs



- No cryogenic air separation unit (ASU)
- No large gas recirculation
- Two interconnected CFB boilers
- Limestone or metal oxide powder as oxygen carrier

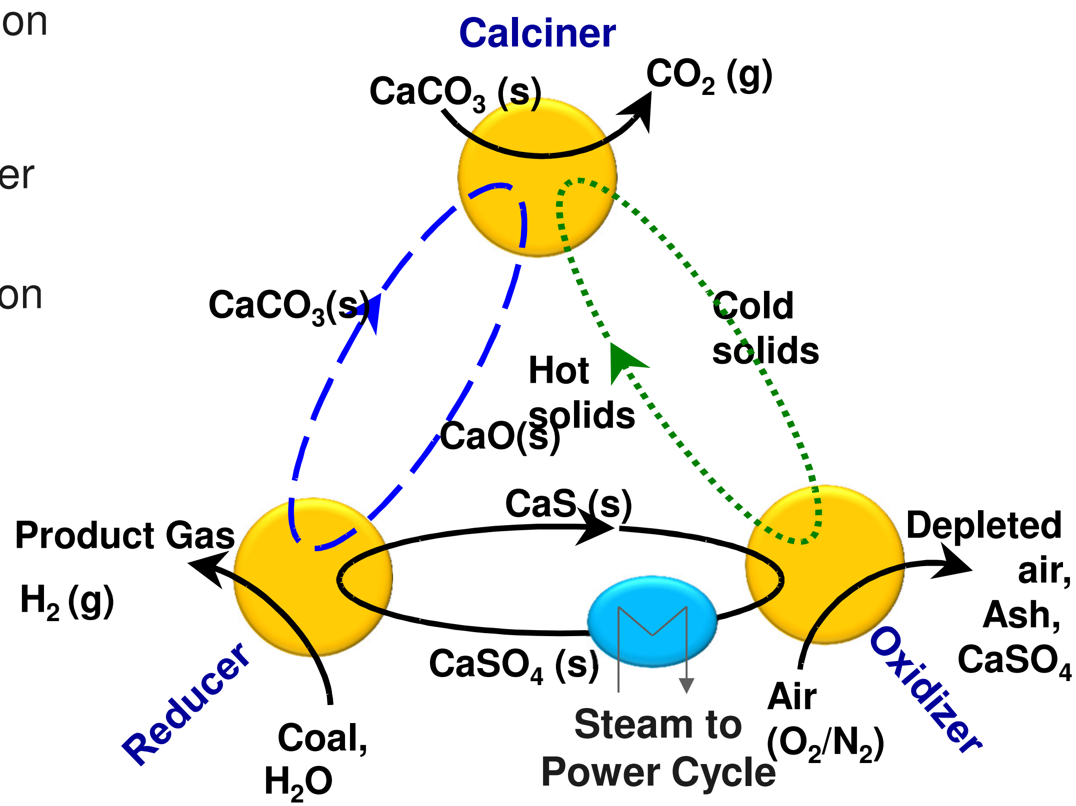
Potential Transformational coal power technology

Chemical Looping Concept – Alstom Limestone-based Process Options:

Option 1 – Chemical looping combustion
- Excess air ($CaSO_4$) to fuel
- Product gas is CO_2
- Heat produces steam for power

Option 2 – Chemical looping gasification
- Excess fuel to air ($CaSO_4$)
- Product gas is Syngas
- No inherent CO_2 capture

Option 3 – Hydrogen production
- Add CaO-$CaCO_3$ to Option 2
- Add calciner
- Product gas is H_2
- Calciner off-gas is CO_2

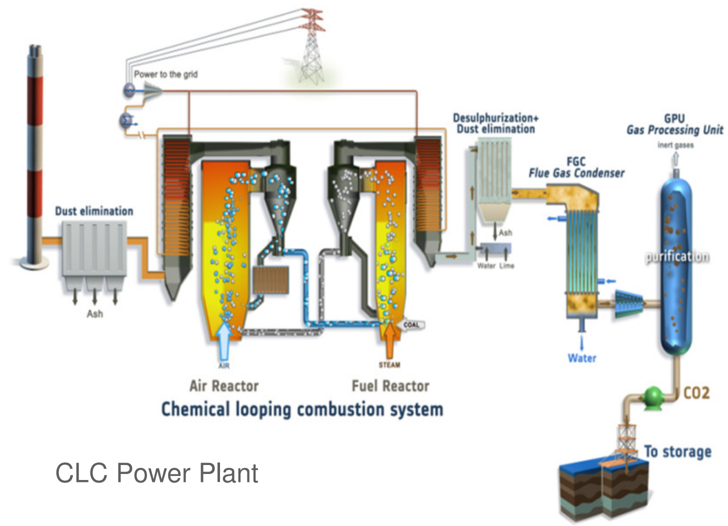


Flexible and Offers Various Options for Hydrocarbon Utilization

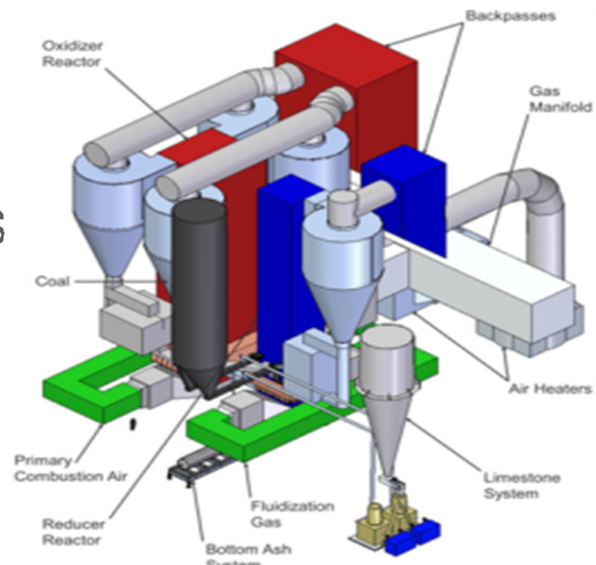
Chemical Looping Plant
Product Vision and Market

- **Product Attributes**:
  - Lowest cost option for coal Power Generation with CCS
  - Lowest energy penalty
  - Fuel flexible
  - Near zero emissions
  - Useful solid ash by-product
  - Application flexible
    - Coal power, syngas, hydrogen
  - Feasible with CFB basis

- **Targets:**
  - Efficiency <10% CCS penalty vs Plant w/o CCS
  - LCoE <30% increase vs. Plant without CCS (<u>stretch target < 20%)</u>
  - CO_2 Capture Cost < \$25/ton (<u>stretch target < \$15/ton</u>)



CLC Power Plant



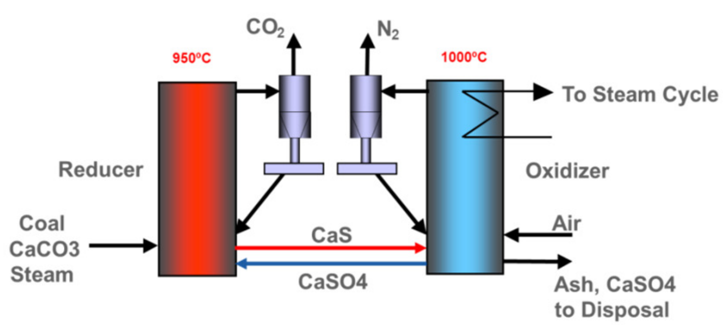
550 MWe Chemical Looping Combustion Steam Generator

Alstom's Chemical Looping Development
Two different oxygen carrier approaches

Limestone based (LCL™)	Metal oxide based (MeOx)

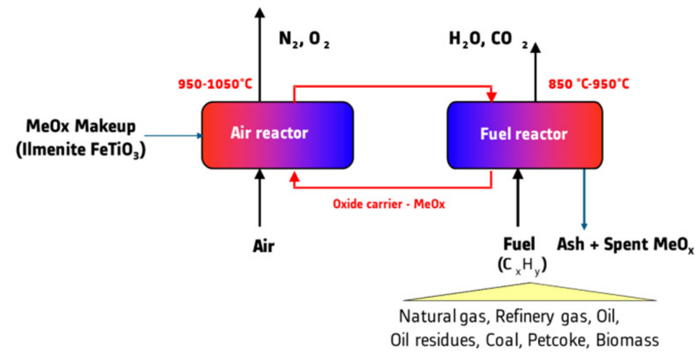
US-DOE co-funded

- Limestone based oxygen carrier - $CaS/CaSO_4$
- "Fast" CFB solids transport
- Same materials in commercial CFBs
- Suitable for solid fuels - coal, petcoke, biomass



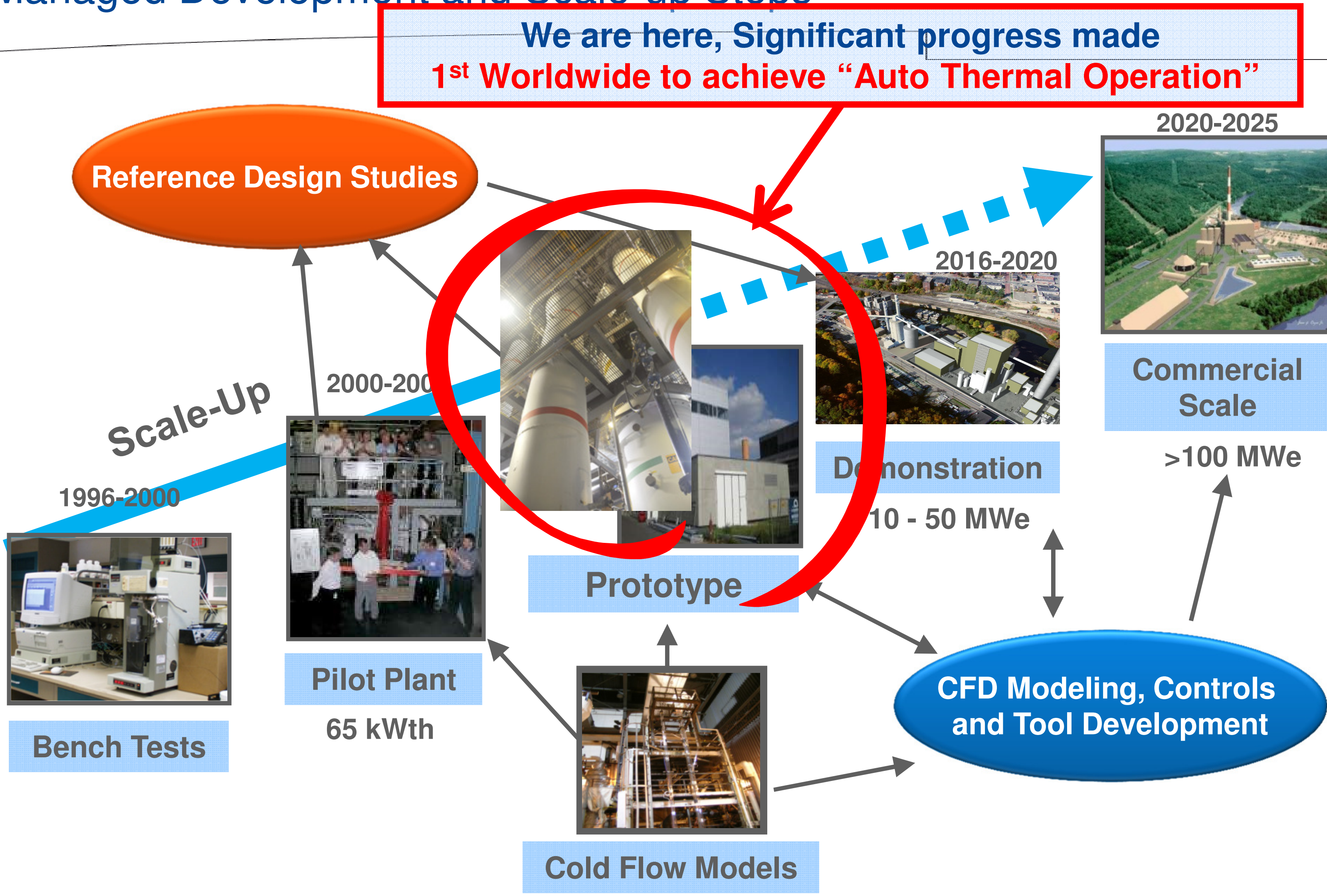
ECLAIR Program–RFCS co-funded

- Metal-based Oxygen Carriers ilmenite ($FeTiO_3$) – iron-titanium ore
- Process based on CFB solids transport
- Carbon stripper for minimizing UBC
- Suitable for gaseous fuels – natural gas



Pursuing two different chemical looping technologies

Alstom - Chemical Looping Process
Managed Development and Scale-up Steps



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Relocation of Alstom Power Plant Labs and Chemical Looping pilot
Dec. 2013 to June 2015

Alstom Clean Energy Lab Inauguration – 21 August 2015

DOE leaders with Alstom staff at CLC pilots

We are shaping the future | ALSTOM

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LCL-C™ process assessment / performance criteria

Gap ID	TECHNICAL GAP
Operability Gap	
1	High Solids Loss
2	Main DipLeg Flushing
3	Stability of Solids Circulation
4	Oxygen Carrier (Sorbent) Activation
5	Sulfur Management
6	Low Reactor Temperatures during Recent Run
Performance Gap	
7	Carbon Carryover to Oxidizer

PERFORMACE IMPACTS

		Commercial Goal	BP2 Current Level Autothermal	BP3 Prototype Success Criteria (significant progress)	BP4 Prototype Success Criteria (significant progress)	Technology Gap
Carbon Conversion Performance						
Carbon Capture in Reducer Gas	(%)	>95	40 to 50	> 70	> 80	1,3,4,6,7
Unburned carbon loss in ash	(%)	< 0.5	1 to 20	< 10	< 5	1,6
CarbonCarryover to Oxidizer	(%)	1	20 to 40	<30	< 20	7
Raw CO2 Stream Oxygen Demand	(%)	<5 (1)	25 to 15	<15	<10	4,6
Sulfur Capture/Retention Performance (2.5% sulfur coal)						
Sulfur capture by limestone	(%)	> 85 (3)	0 to 50%	> 50	> 70	4,5
Solids Transport Performance						
Solids Loss Rate (max at full load)	(lb/hr)	as req'd for deSulf	variable to 2000	< 1000	< 500	1,2,3
Minimum maintainable Reducer Temp	(degF)	1850	1550 to 1750	1650 to 1800	1650 to 1850 (2,4)	6
Oxidizer - Reducer Temperature	(degF)	100	300 to 200	< 250	< 200	6
Main DipLeg Flushing		eliminate	frequent with coal	occasional with coal	rare with coal	2,3

Notes:
1. Allows CO for deSulfurization. Handeled via GPU recycle or O2.
2. Allows tradeoff for deSulfurization vs carbon loading (i.e. solids residence time).
3. Remainder via GPU recycle and Oxidizer gas cleanup.
4. Coal dependent.

SuccessCriteria.xls
11 Nov 2013
rev 14 Feb 2014

How can we improve performance?

Project description

- **Objective:** To further develop a coal-fired LCL-C™ process that can produce a higher purity flue gas stream and achieve an improved overall performance while achieving greater than 90% CO_2 removal at less than 35% increase in the Levelized Cost Of Electricity (LCOE)
- The Oxygen Demand (OD) targeted is OD < 5% (enhanced LCL-C™) and OD < 1% (polishing stage)
- 3 technical approaches will be followed:
  - 1st technical approach: Improved LCL-C™ Oxygen Carrier
  - 2nd technical approach: Oxy-combustion downstream of the reducer
  - 3rd technical approach: Gas processing Unit with reducer product gas recycle

Oxygen demand definition: percentage of oxygen to be supplied to the product gas (e.g. by ASU) to achieve complete combustion of such product gas with respect to the stoichiometric oxygen required for complete combustion of the fuel

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Identified technical approaches to an improved gas purity

1. Improved LCL-C™ oxygen carrier/process performance (syngas, volatiles)

2. Oxy-combustion downstream of reducer:

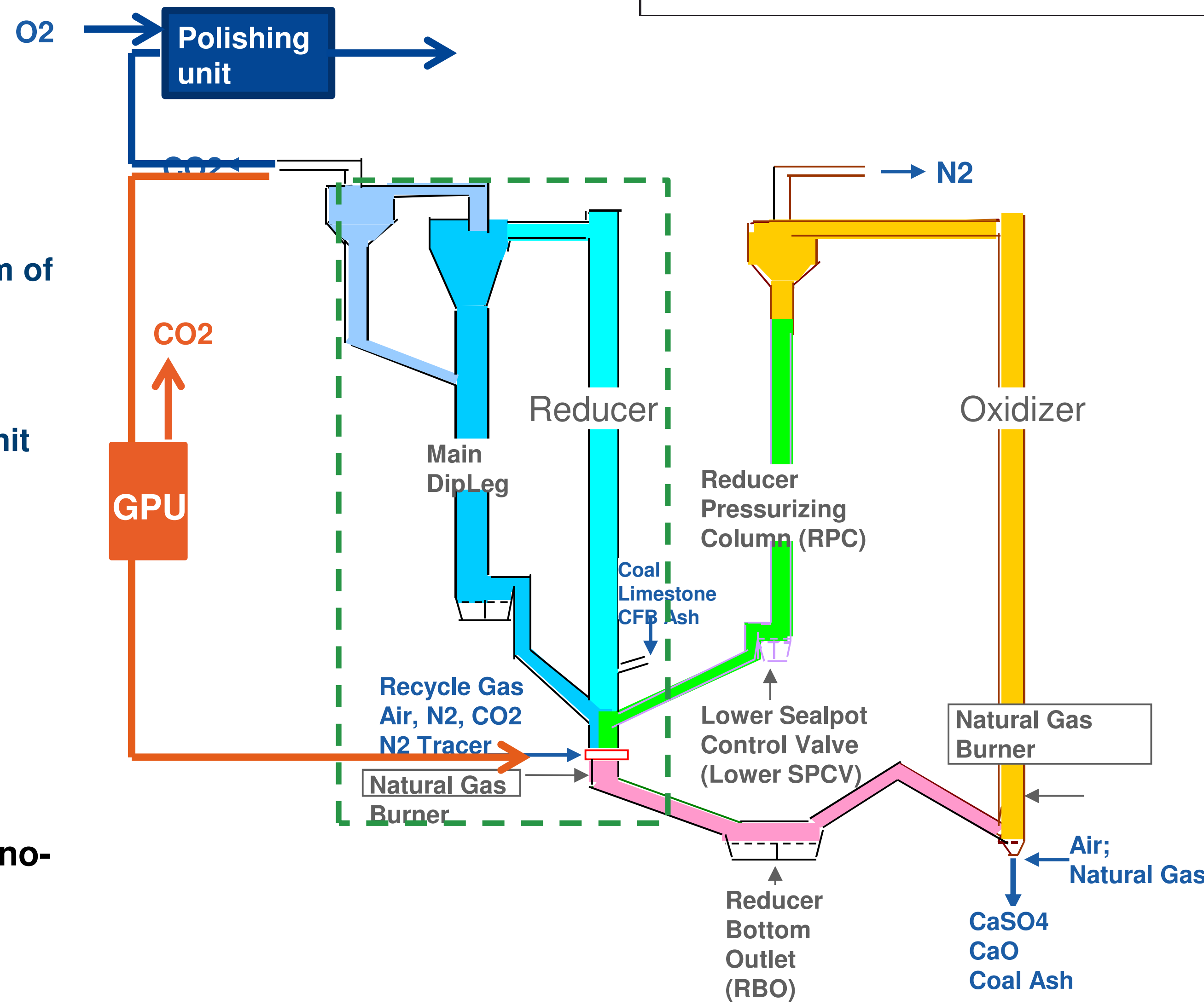
Oxygen injection

Secondary chemical looping unit (polisher)

3. Gas Processing Unit with reducer gas recycling

4. Any combination of the 3 approaches above

5. Downselect based on techno-economic analysis



1st technical approach: improved LCL-C™ oxygen carrier

Assessment of the $CaSO_4$/CaS carrier

- Thermodynamics
- Oxygen capacity
- Thermochemistry
- Kinetics / selectivity
- Sulfur-containing compounds emissions

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Thermodynamics of LCL-C™

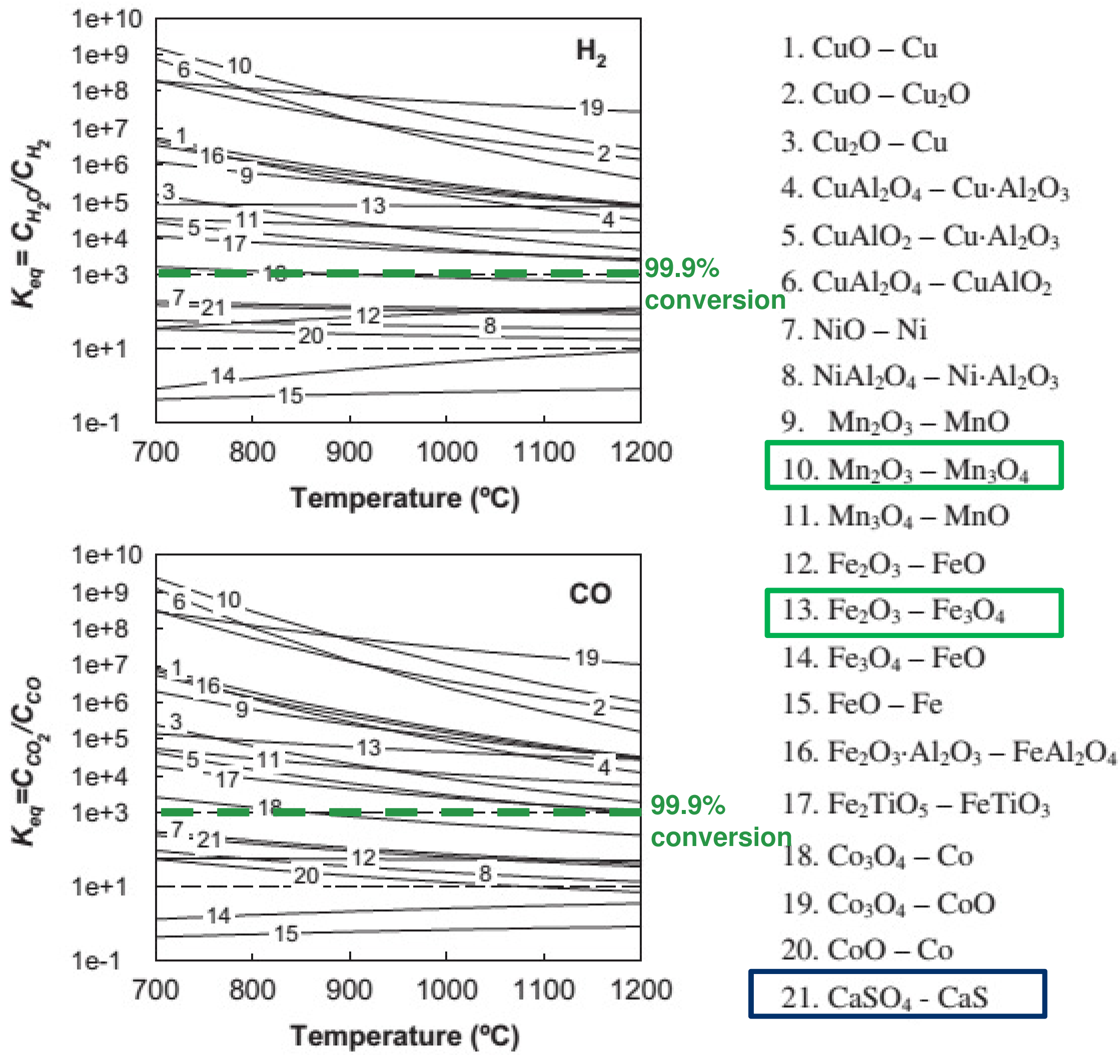

Fig. 3. Equilibrium constant, K_{eq}, for the reduction reaction with H_2 and CO with different redox systems.

CaS-$CaSO_4$: 98.5% conversion of syngas thermodynamically possible

Adanez et al. (2012)

Oxygen capacity for LCL-C™ is large but…

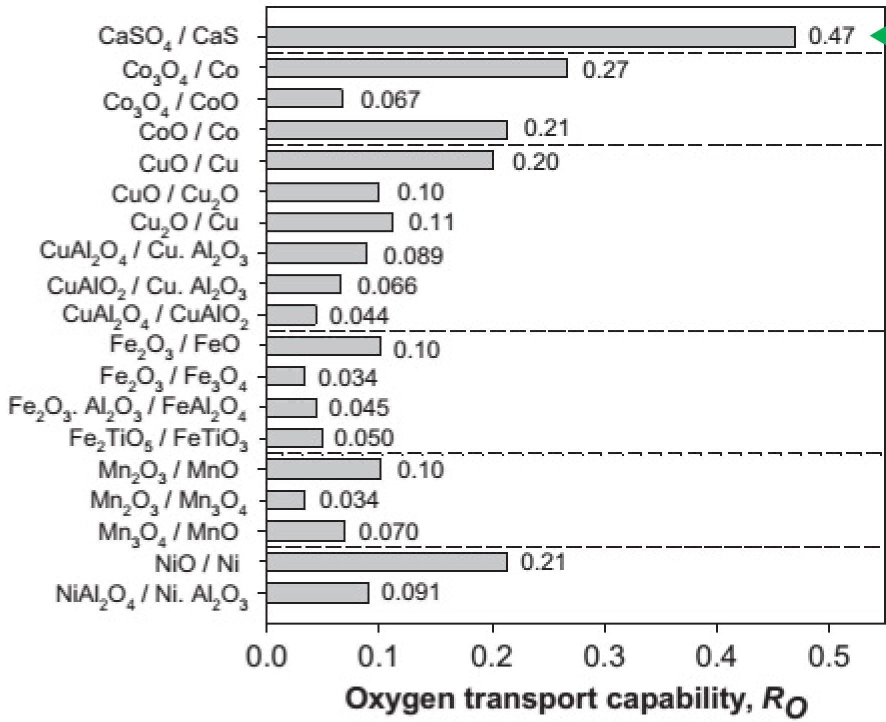

Fig. 4. Oxygen transport capability, R_O, of different redox systems.

Adanez et al. (2012)

100% / pure anhydrite

$$R_O = \frac{m_o - m_r}{m_o}$$

Test 2 solids composition

Mass fraction (fully oxidized)		
CaO	0.141	kg/kg
CaSO4	0.254	kg/kg
CaCO3	0.000	kg/kg
MgO	0.038	kg/kg
SiO2	0.227	kg/kg
Al2O3	0.151	kg/kg
Fe2O3	0.189	kg/kg
Sum	1.000	kg/kg

Mass fraction (fully reduced)		
CaO	0.141	kg/kg
CaS	0.135	kg/kg
CaCO3	0.000	kg/kg
MgO	0.038	kg/kg
SiO2	0.227	kg/kg
Al2O3	0.151	kg/kg
Fe2O3	0.189	kg/kg
Sum	0.881	kg/kg

Beware of dilution effect!

$$R_{OC} = x_{OC} R_O$$

Ro	11.94%

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Oxygen capacity increase for LCL-C™

- The concept of Steam Activation Heat Exchanger (SAHE) has already been demonstrated under DE-FC26-03NT41866
- Steam injection is first used to hydrate, crack and weaken the CaS or $CaSO_4$ shell which surrounds the underlying CaO.
- The particle is then pneumatically transported to hit a hard target surface in the Impactor to further crack the particles and expose more active CaO
- Active CaO can play a role in increasing gasification / combustion kinetics
- Active CaO can plan a role in increasing sulfur (H_2S) re-capture kinetics

Enhanced utilization of the OC oxygen content needs to be assessed

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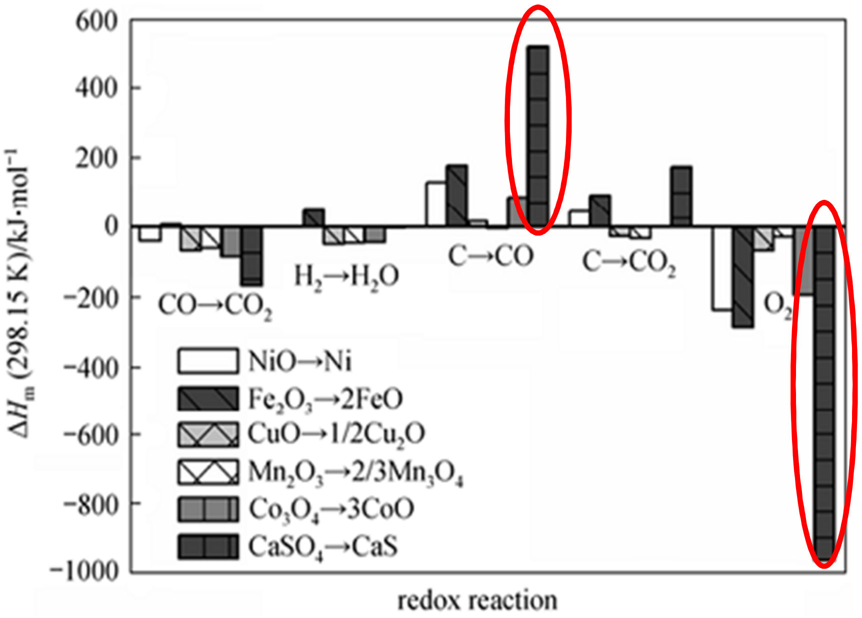
Thermochemistry: heat management for LCL-C™



Guo et al. (2011)

Redox system	ΔH_r^0 (kJ/mol gas or C)				
	CH_4	H_2	CO	C	O_2
$CaSO_4/CaS$	158.6	−1.6	−42.7	86.9	−480.5
Co_3O_4/Co	107.9	−14.3	−55.4	61.6	−455.1
Co_3O_4/CoO	−16.8	−45.5	−86.6	−0.8	−392.7
CoO/Co	149.5	−3.9	−45.0	82.4	−475.9
CuO/Cu	−178.0	−85.8	−126.9	−81.4	−312.1
CuO/Cu_2O	−236.6	−100.4	−141.6	−110.7	−282.8
Cu_2O/Cu	−119.5	−71.1	−112.3	−52.1	−341.4
$CuAl_2O_4/Cu\cdot Al_2O_3$	282.2	29.3	−11.8	148.7	−542.2
$CuAlO_2/Cu\cdot Al_2O_3$	−24.1	−47.3	−88.4	−4.4	−389.1
$CuAl_2O_4/CuAlO_2$	588.5	105.9	64.7	301.9	−695.4
Fe_2O_3/Fe_3O_4	141.6	−5.8	−47.0	78.4	−472.0
Fe_2O_3/FeO	318.4	38.3	−2.8	166.8	−560.3
$Fe_2O_3\cdot Al_2O_3/FeAl_2O_4$	−62.3	−56.8	−98.0	−23.5	−370.0
$Fe_2TiO_5/FeTiO_3$	106.5	−14.6	−55.8	60.9	−454.4
Mn_2O_3/MnO	−48.0	−53.3	−94.4	−16.4	−377.1
Mn_2O_3/Mn_3O_4	−396.6	−140.4	−181.6	−190.7	−202.8
Mn_3O_4/MnO	126.3	−9.7	−50.8	70.8	−464.3
NiO/Ni	156.5	−2.1	−43.3	85.9	−479.4
$NiAl_2O_4/Ni\cdot Al_2O_3$	158.6	−1.6	−42.8	86.9	−480.4

CaS oxidation: largely exothermic reaction

=> $CaSO_4$ reduction with products of gasification is also largely endothermic reaction

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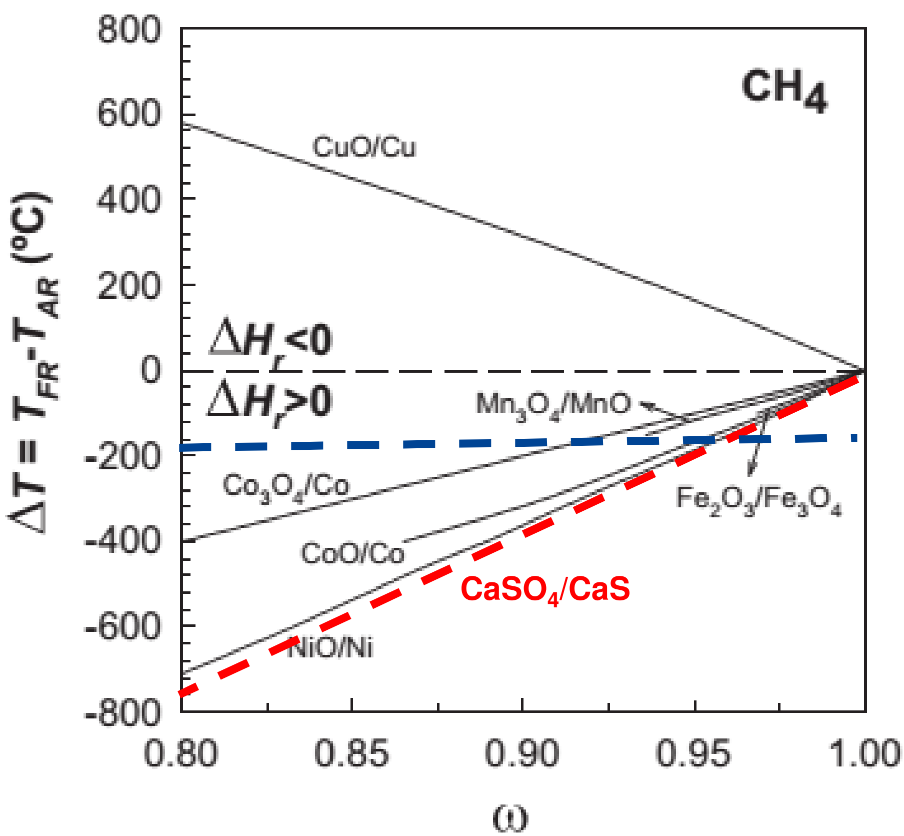
Thermochemistry => impact solids circulation rate



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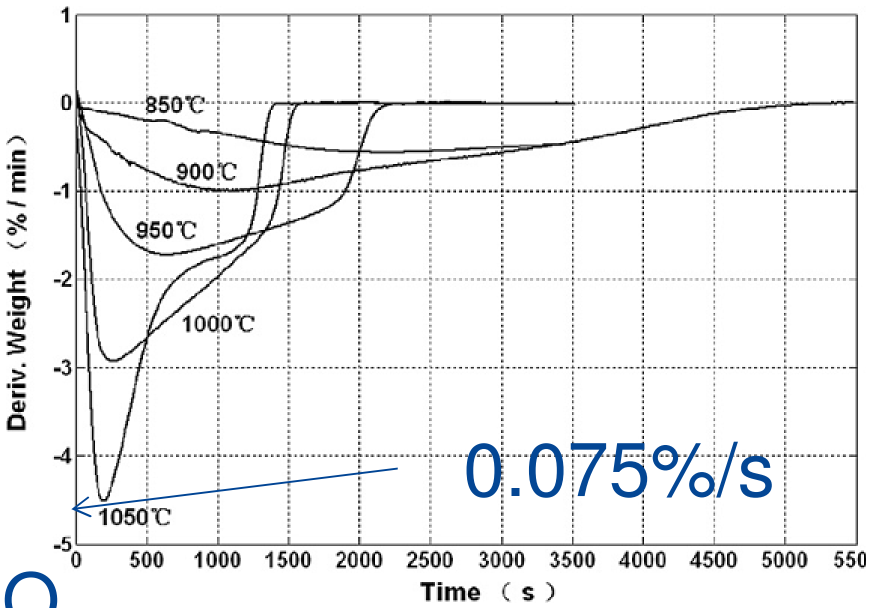
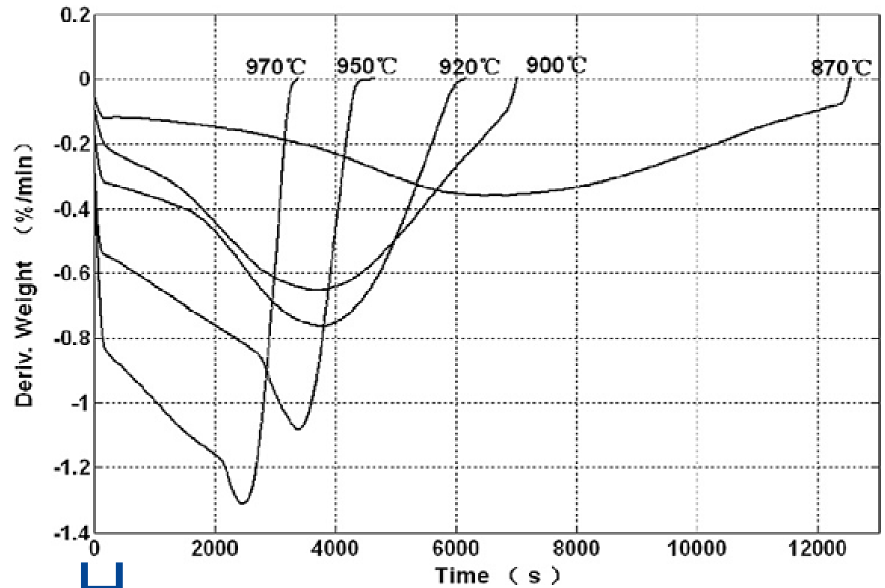
In CL-C, oxygen carriers with highly endothermic reactions with C will dictate achievable solid conversions

Kinetics of $CaSO_4$/CaS reduction



CO



H_2

Shen et al. (2008)

	Fe2O3/ Fe3O4	NiO	CuO/Cu2O	Mn3O4/MnO	CoO	CaSO4/CaS
Reduction kinetics/ Reactivity (w/ syngas)	~	+	+	~	-	~

Guo et al. (2012)

There are opportunities to improve reduction kinetics

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Reactivity/Conversion/selectivity

For iron ore

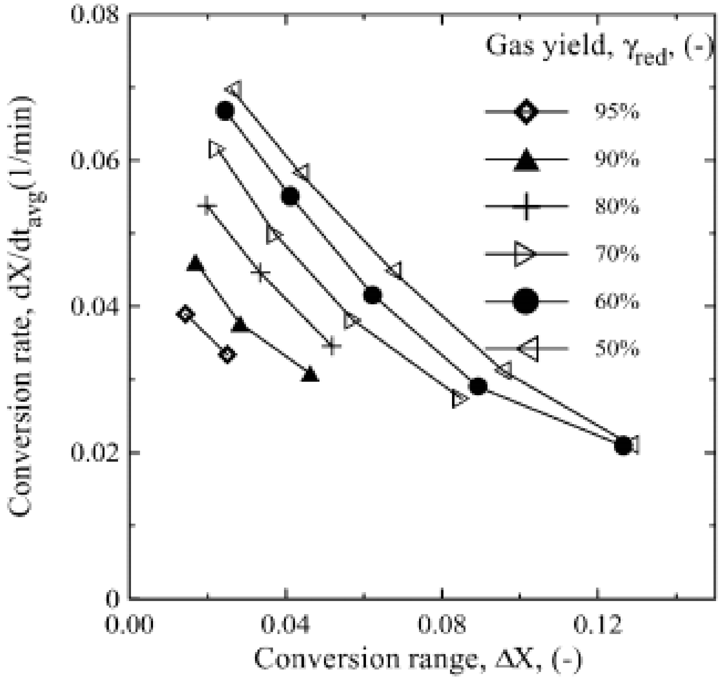

Fig. 10. The average rate of reduction, dX/dt_{avg}, as a function of the conversion range, ΔX, for different degrees of methane yield to carbon dioxide, γ_{red}.

Mattisson et al. (2001)

A kinetics study for preferred LCL-C^{TM} enhanced oxygen carriers should provide an understanding of the mapping between dX/dt, ΔX, and yield

=> **Higher yield and conversion at lower reducer T are targeted**

Side reactions: sulfur containing species

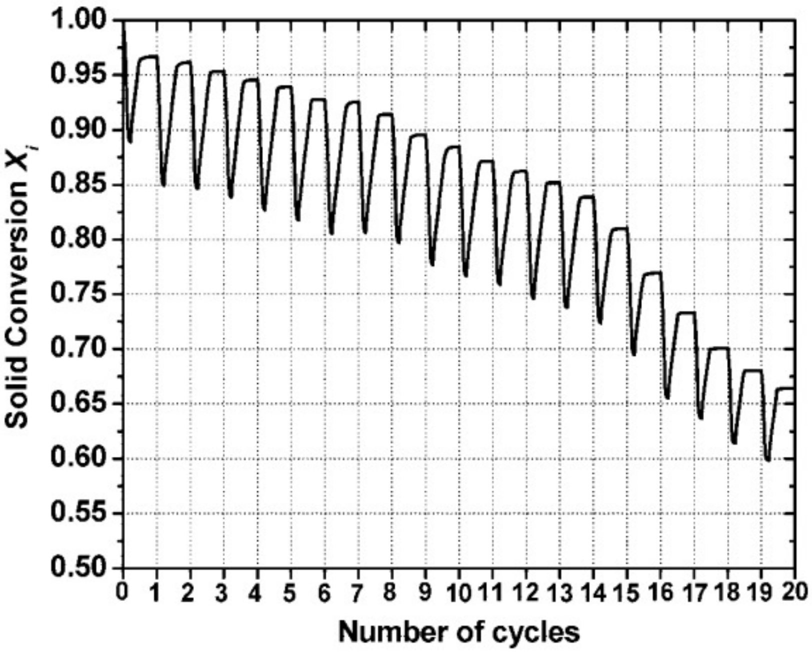

Figure 5. Variation of the oxygen carrier conversion as a function of the number of cycles.

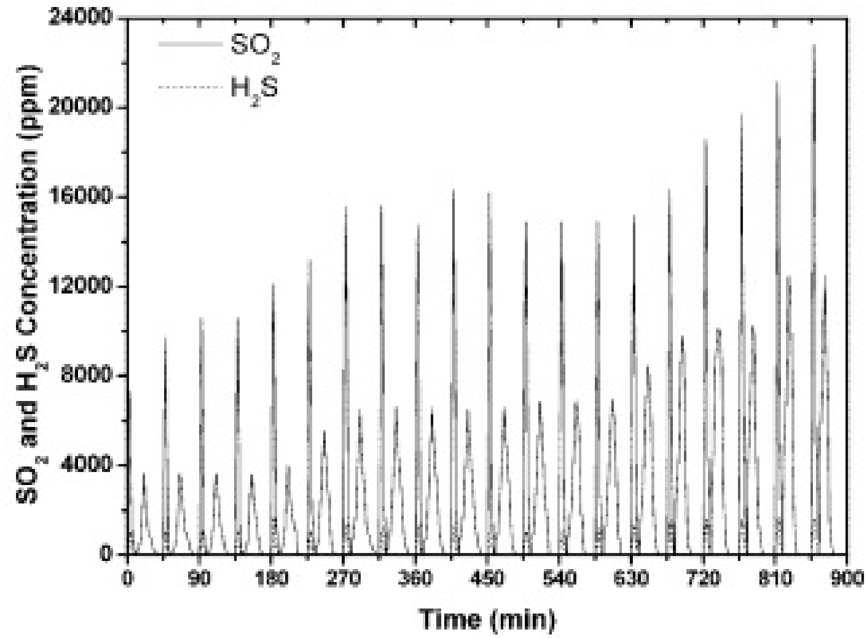

Figure 3. Outlet SO_2 and H_2S concentrations as a function of time during the 20 cyclic tests.

Song et al. (2008)

Loss of OC capacity by sulfur loss will reduce gas yield and purity over time. Sulfur will need to be recaptured to prevent emissions

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Summary on planned 1st technical approach (LCL-C™ sorbent improvement)

1. Screening of oxygen carrier blends (in collaboration with University of North Dakota – UND, focus on low cost sorbents) under conditions specific to LCL-C™
   - Improved thermodynamics of syngas oxidation
   - Improved kinetics of syngas oxidation/volatile compounds
   - Reduced exothermicity/ endothermicity for
   - Improved SO_2/H_2S capture or reduced release

➡ **Blends with Mn-based, Fe-based ores with LCL-C™ carrier as well and CaO blends (make up) represent opportunities**

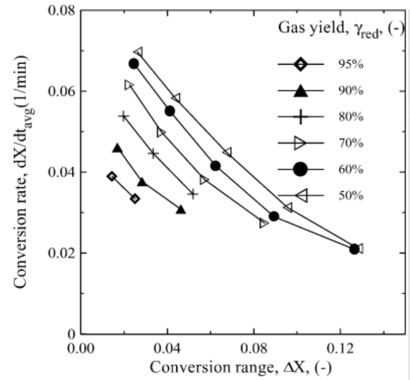
2. Enhanced performance of LCL-C™
   - Improved use of LCL-C™ carrier oxygen capacity
   - Reduce sulfur loss and cyclic degradation

➡ **Steam activation of the LCL-C™ sorbent**

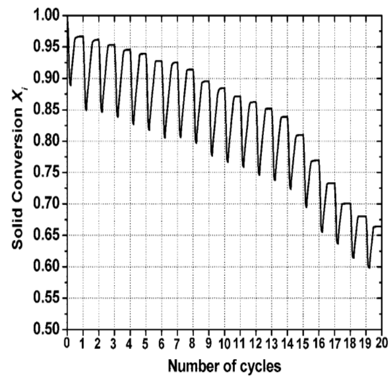
Planned execution: 1st technical approach, Step 1 collaboration with UND/Envergex for bench-scale testing

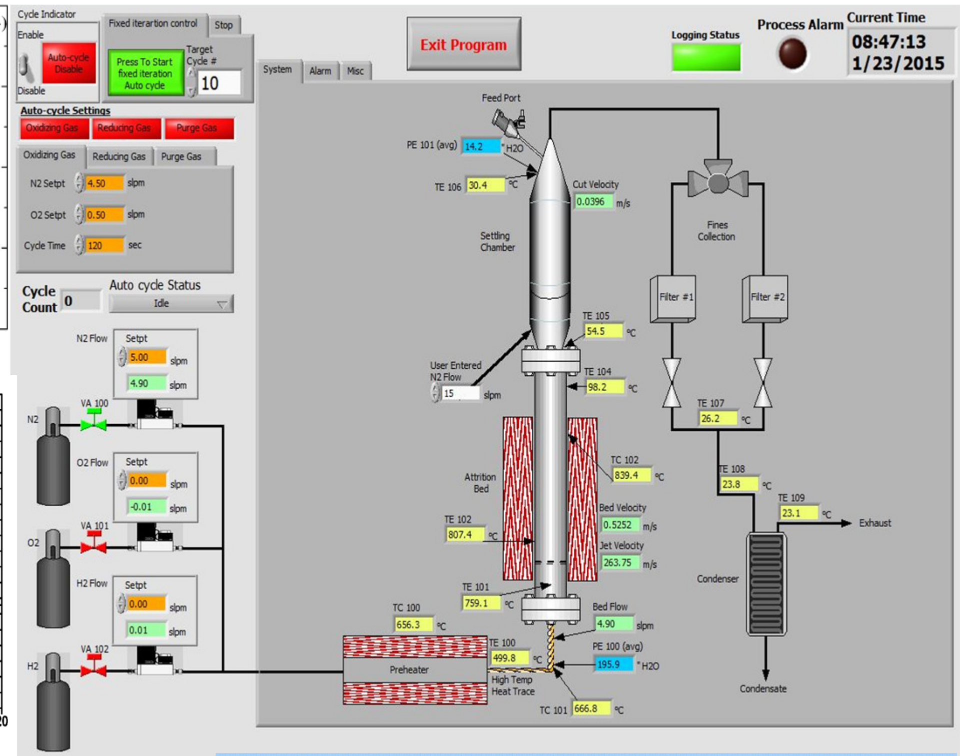
Objective: screening of oxygen carrier blends and their performance in terms of:

- Reactivity / selectivity

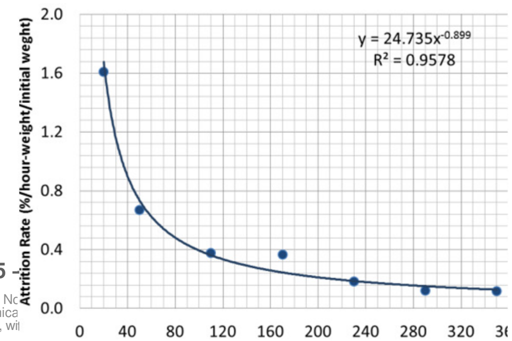


- Cyclability





- Attrition
- Physical properties
- Morphology



Fully automated bench scale facilities

=> ~ 20 tests scheduled

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Planned execution: 1st technical approach, Step 1, 2, and 3

STEP 1

Bench Tests

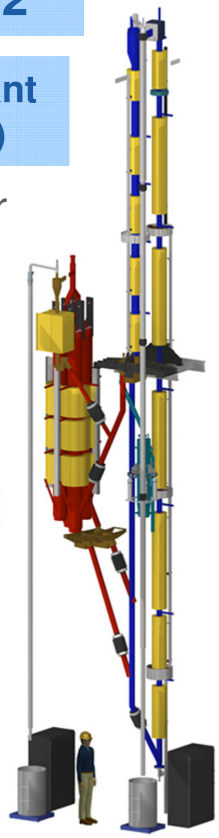
- 50g (batch)
- Gaseous fuels only

Univeristy of North Dakota labs

STEP 2

Pilot Plant (PSTF)

- ~5000lb/hr
- Gas and solid fuels

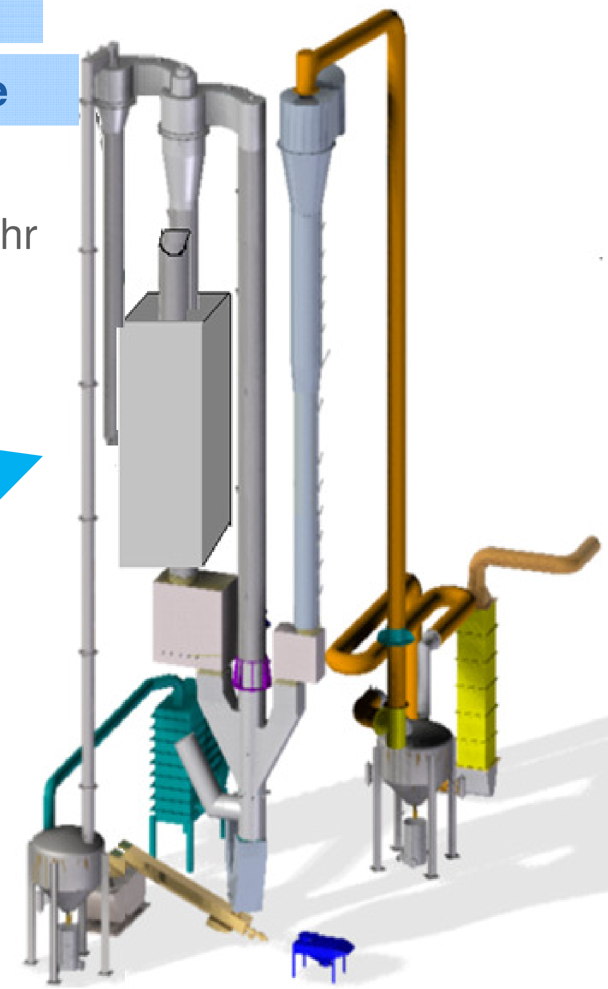


Alstom‘s Clean Energy Labs, CT

STEP 3

Prototype

- Up to 200,000lb/hr
- Gas and solid fuels



Alstom‘s Clean Energy Labs, CT

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2nd technical approach: oxy-combustion downstream of reducer

Assessment of gas polishing options

- Brute force oxygen injection (TEA analysis only)
- Screening of oxygen carriers with deep polishing capabilities on gas product
  - Leverage Alstom's past work with metal oxides
  - Screening of oxygen carriers with Chemical Looping Oxygen Uncoupling (CLOU)

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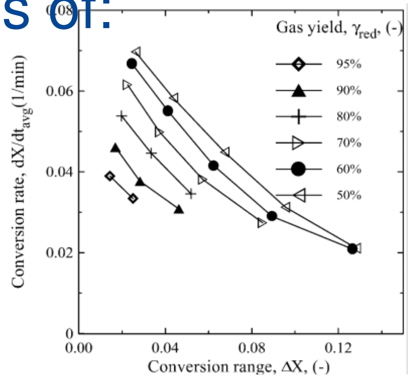
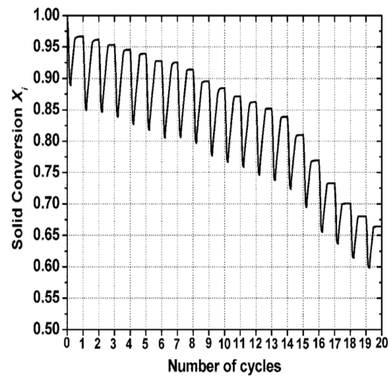
Planned execution: 2st technical approach, Step 1 collaboration with UND/Envergex for bench-scale testing

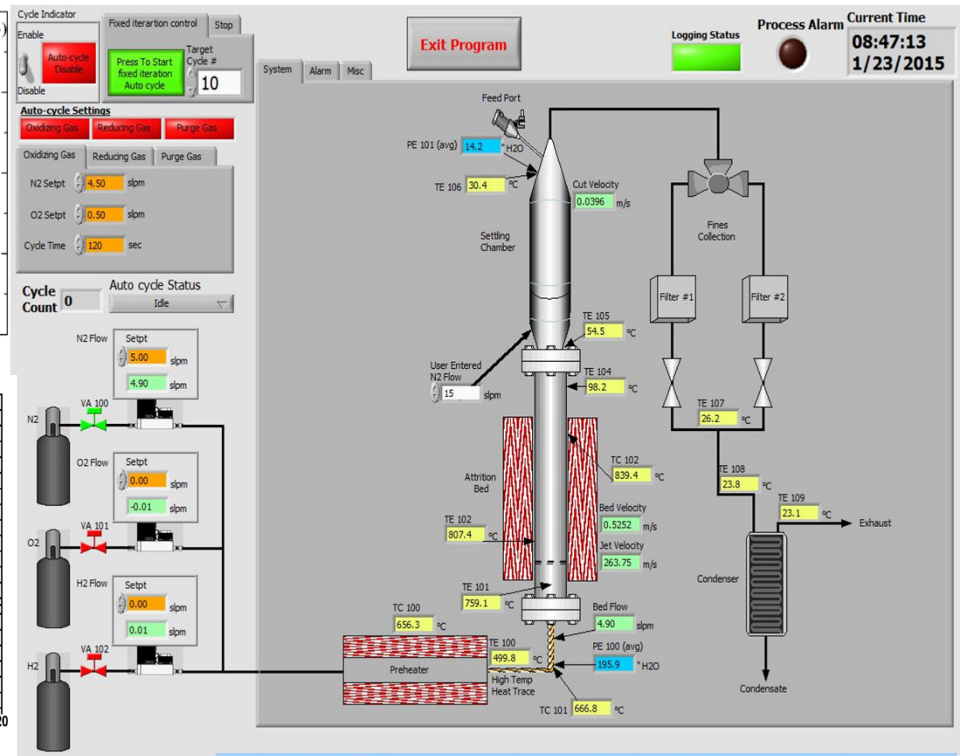
Objective: screening of polishing oxygen carrier (CLOU) and their performance in terms of:

- Reactivity / selectivity

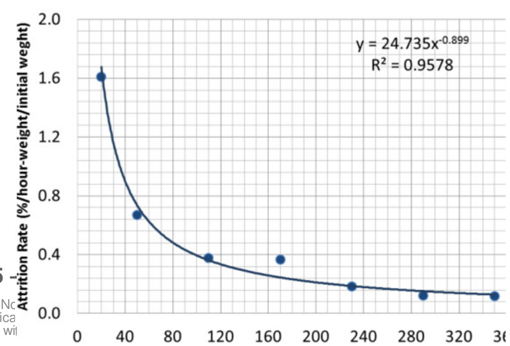


- Cyclability





- Attrition
- Physical properties
- Morphology



Fully automated bench scale facilities

=> ~ 40 tests scheduled

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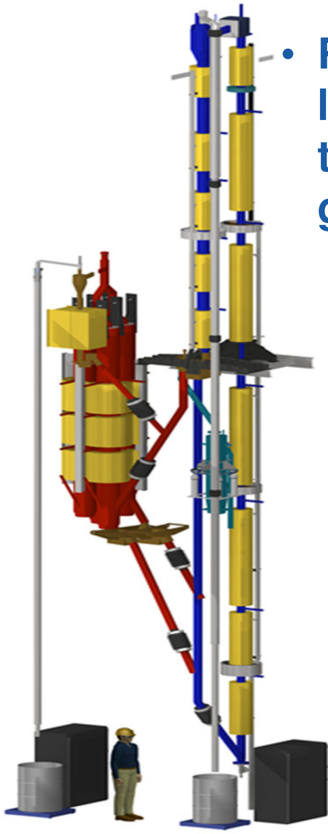
Planned execution: 2st technical approach, Step 2: scale up to pilot scale

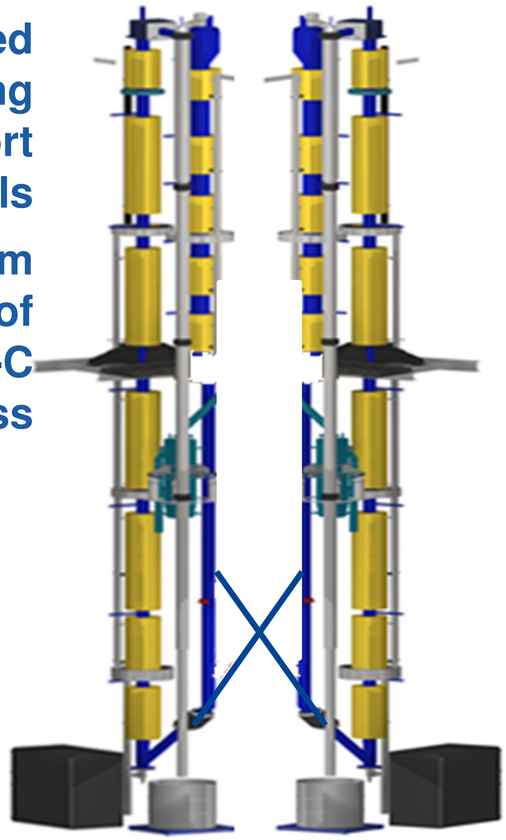
Objective: modify existing pilot plant to demonstrate process/oxygen carrier performance/stability under polishing process conditions



- **Reducer box for longer residence time required for gasification**

- **Interconnected circulating fluidized/transport beds for gas fuels**

=> Downstream configuration of existing LCL-C process



Enhanced LCL-C (solid fuel configuration

Enhanced LCL-C (gas fuel configuration)

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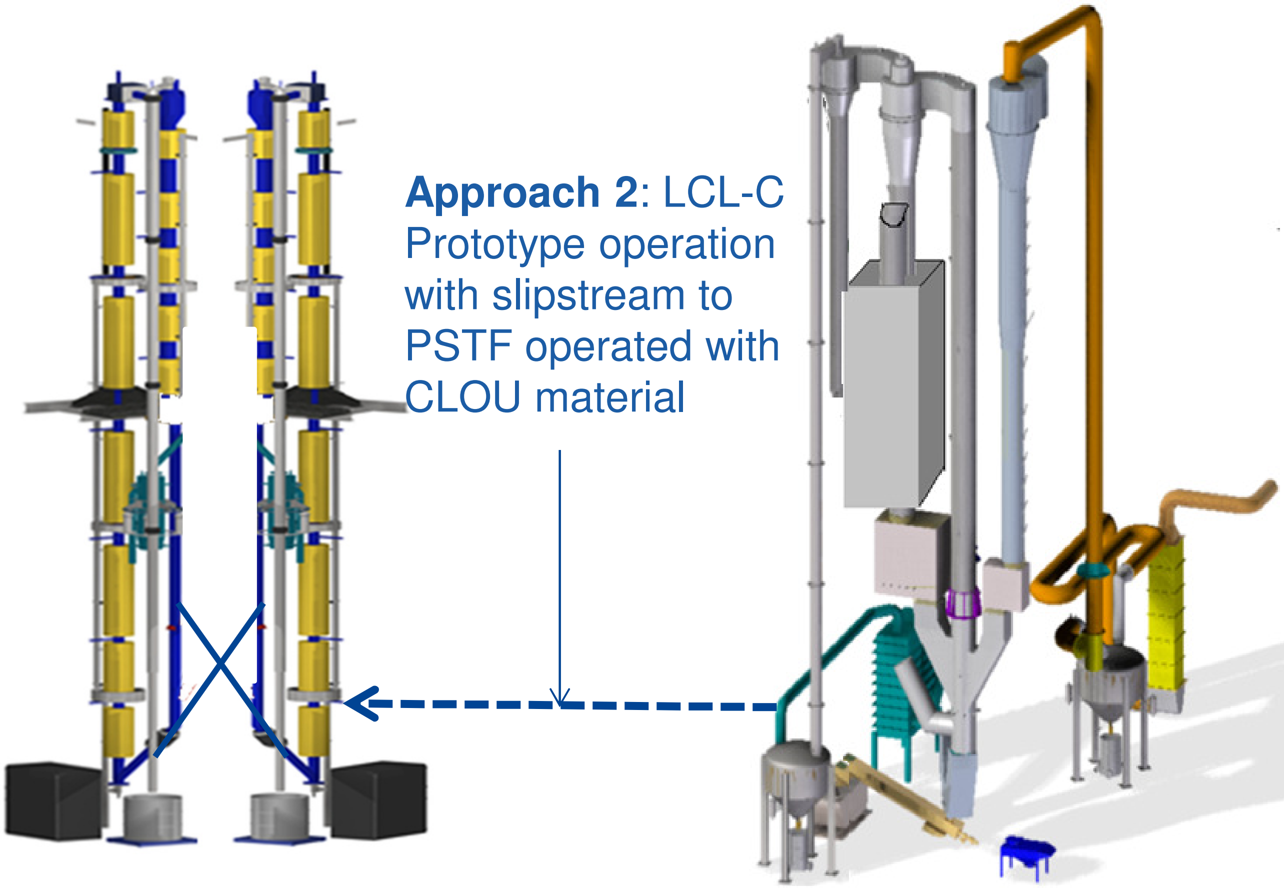
Planned execution: 2st technical approach, Step 3: performance validation on 3-MWth Prototype slipstream

Objective: demonstrate reduced oxygen demand at the Prototype scale using the best configuration identified during previous phase



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3rd technical approach: reducer gas recycling

Assessment of gas recycling concept (simulation only)

- Aspen Plus modeling of the Gas Processing Unit developed by Alstom for CO_2 capture/purification post Oxy-combustion
- Implementation of the thermodynamics (VLEs) of syngas and methane into the existing model to assess process entitlement in terms of gas separation
- Results to feed into Techno Economic Analysis to benchmark against other approaches or combinations

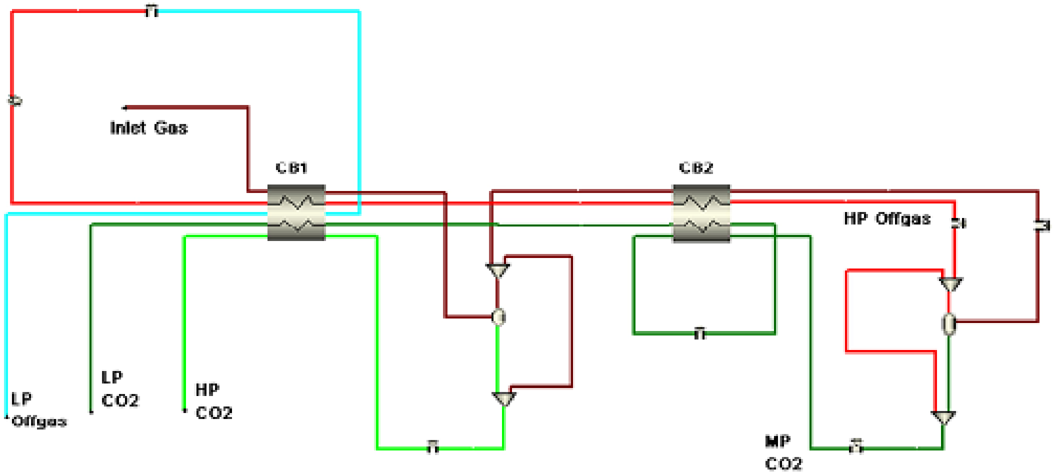

Figure 1. Process flow diagram for cold box in Aspen.

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AGENDA

- **Background**
  - What's Chemical Looping?
  - Why Chemical Looping?
  - Alstom's Chemical Looping Roadmap
- Technical approach to improved Gas Purity
- Project objective
- Project structure
- Project schedule
- Project budget
- Project Management Plan including Risk Management

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Project Objective and success criteria

- **Objective:** To further develop a coal-fired LCL-C™ process that can produce a higher purity flue gas stream and achieve an improved overall performance while achieving greater than 90% CO_2 removal at less than 35% increase in the Levelized Cost Of Electricity (LCOE)

Tasks	Criteria
1. Project Management and Planning	(1) Successful completion of the project on time and on budget (2) On-time delivery of project deliverables
2. Bench-scale Process investigation and Prototype Support	(1) Oxygen demand in product gas reduced below 5% (for mixed oxygen carriers combinations) or below 1% (for polishing oxygen carriers) at pilot scale (2) Generation of sufficient data for scale-up study to prototype scale with a targeted oxygen demand of 1%
3. Prototype Testing	(1) Successful installation of modifications and shakedown of facility (2) Generation of sufficient results from the integrated 3-MW_{th} testing to assess the key process factors for oxygen demand and carbon capture
4. Techno-Economic Analysis (TEA)	(1) TEA based on available results does not negate feasibility

Oxygen demand definition: percentage of oxygen to be supplied to the product gas (e.g. by ASU) to achieve complete combustion of such product gas with respect to the stoichiometric oxygen required for complete combustion of the fuel

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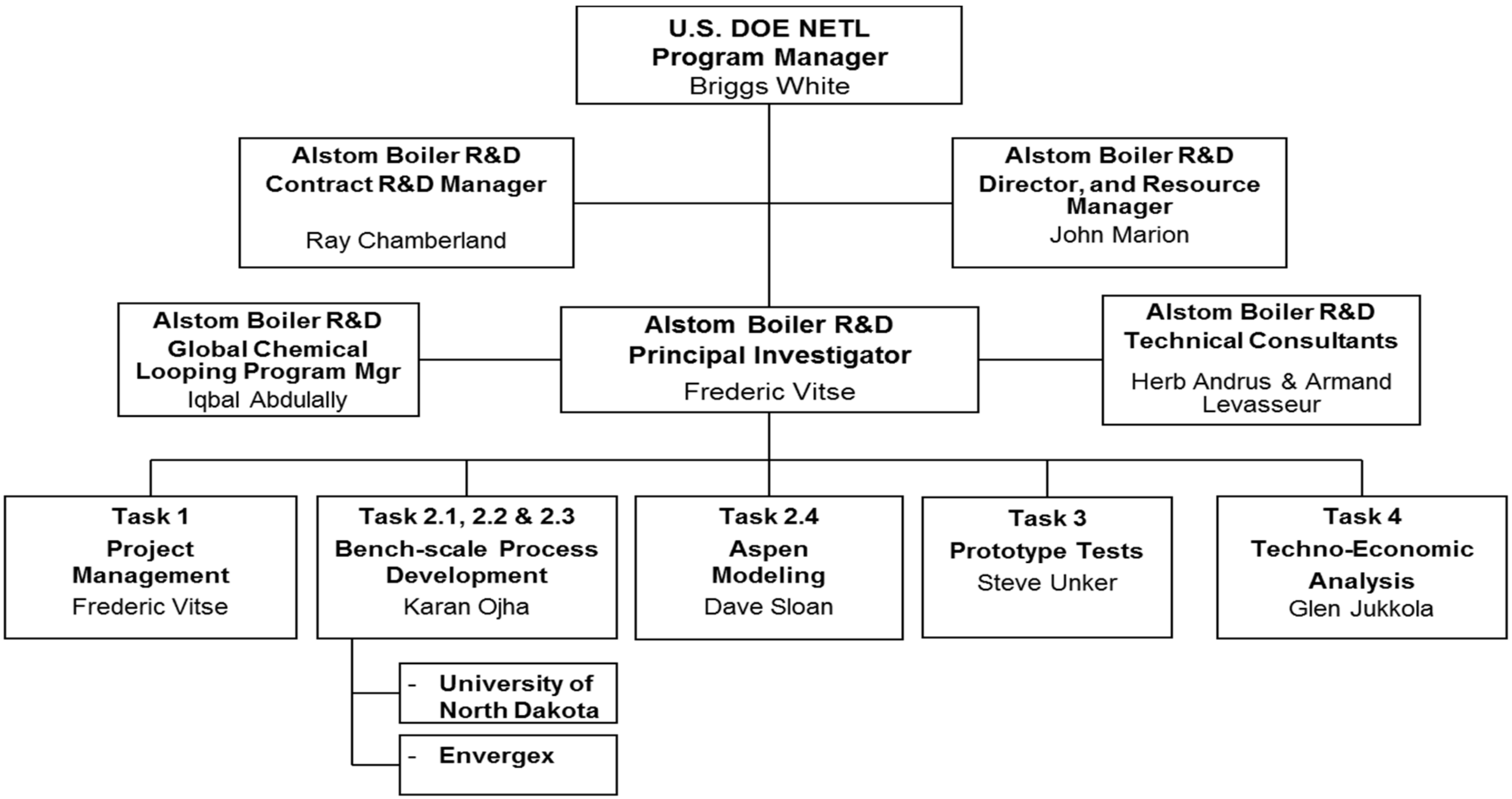
FE25073 – Organization chart



Expert resources (with the experience of LCL-C and LCL-G) are committed to this project

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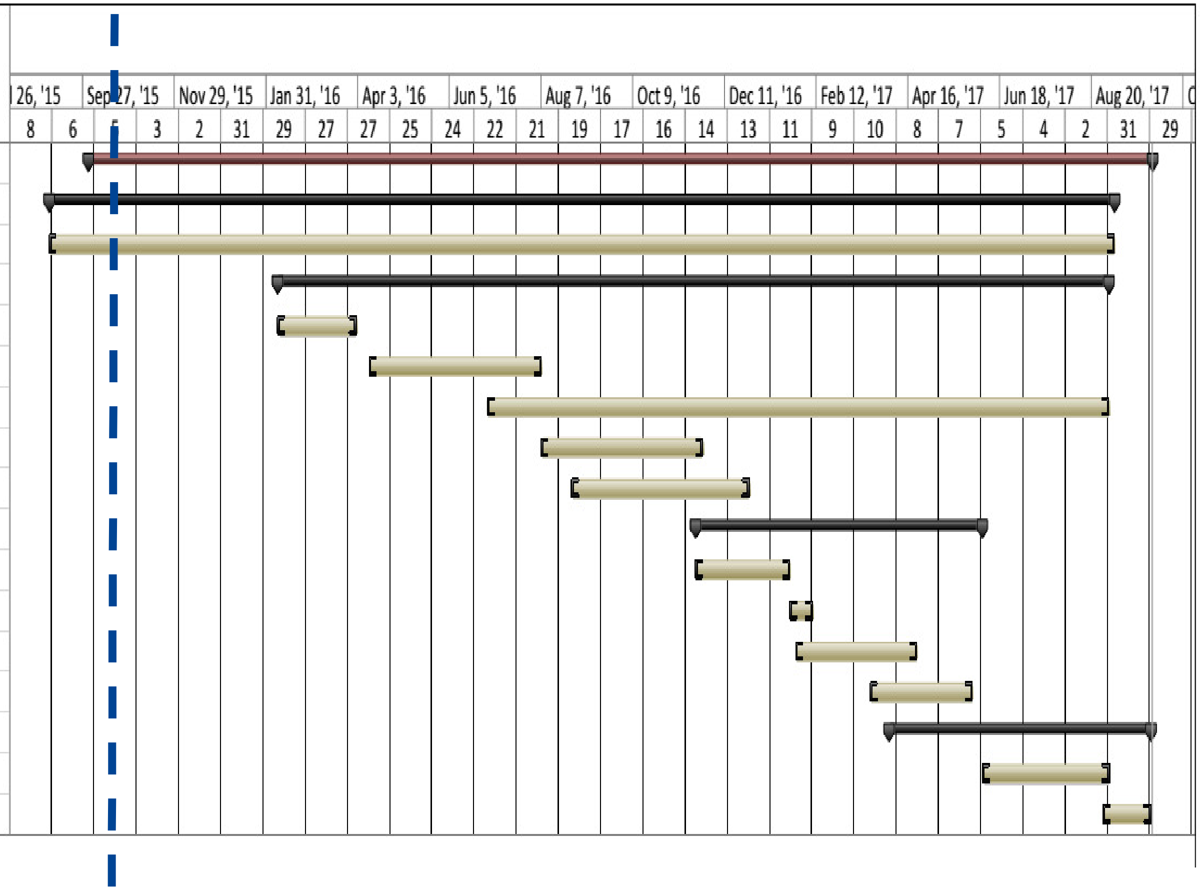
FE25073 –Schedule

ID	Task Mode	Task Name	Start	Finish
1		**DE-FOA001247**	**Thu 10/1/15**	**Sat 9/30/17**
2		**Project Management and Engineering**	**Fri 9/4/15**	**Mon 9/4/17**
3		Project Management and Planning	Fri 9/4/15	Mon 9/4/17
4		**Bench Scale Testing**	**Mon 2/8/16**	**Thu 8/31/17**
5		*Bench Test Rig Modifications and Prep*	Mon 2/8/16	Fri 4/1/16
6		*UND/Envergex bench tests*	Mon 4/11/16	Sun 8/7/16
7		*PSTF upgrade and testing*	Fri 7/1/16	Thu 8/31/17
8		*Test data Processing*	Sun 8/7/16	Sat 11/26/16
9		*Reporting*	Sun 8/28/16	Tue 12/27/16
10		**Prototype testing**	**Mon 11/21/16**	**Mon 6/5/17**
11		*3 MW Prototype E & M*	Mon 11/21/16	Tue 1/24/17
12		*LCL-C tests*	Wed 1/25/17	Thu 2/9/17
13		*Prototype test data post-processing*	Sun 1/29/17	Fri 4/21/17
14		*Reporting*	*Tue 3/21/17*	*Mon 5/29/17*
15		**Final reporting**	**Mon 4/3/17**	**Fri 9/29/17**
16		Techno-Economic Analysis	Tue 6/6/17	Fri 9/1/17
17		Final report	Mon 8/28/17	Fri 9/29/17



- **Project started on September 4^{th} 2015. 1 budget period, 2-yr contract**
- **Ongoing preparation of bench-scale testing equipment and test campaigns**

Milestones and Deliverables

Milestone	Task	Description	Deliverables	Planned Completion	Verification Method
1	1.1	Updated Project Management Plan	1	10/4/2015	Plan file
2	1.1	NEPA Questionnaire	2	10/31/2015	Report File
3	1.1	Project Kickoff Meeting		11/30/2015	Presentation File
4	2.1	Bench Scale tests: Reaction kinetics and side reactions		6/30/2016	Test Data File
5	1.2	Annual Review Meeting	3	10/4/2016	Presentation File
6	2.2	100mm PSTF: Shakedown and carrier enhancement testing		11/31/2016	Test Data File
7	2.2	100mm PSTF test: Product Gas Polishing material integration test		5/31/2017	Test Data File
8	3.2	3MW Prototype Test		7/31/2017	Test Data File
9	4	Updated 550 Mwe LCL-C Technoeconomic Study		8/30/2017	Report File
10	1.1	Project Closing Meeting	4	10/4/2017	Presentation File
11	2.3	Test Program Summary	5	11/31/2017	Presentation File
12	1.2	Final report	6	12/4/2017	Report File
13	1.2	Quarterly progress reports	7	Quarterly	Report File

Project schedule: next immediate steps

- Select/collect metal ores and limestones for bench scale testing

- Kick-off bench scale equipment upgrade at UND

- Kick-off process simulation effort (GPU modeling)

- Initiate reactor design for PSTF upgrade (CLOU material)

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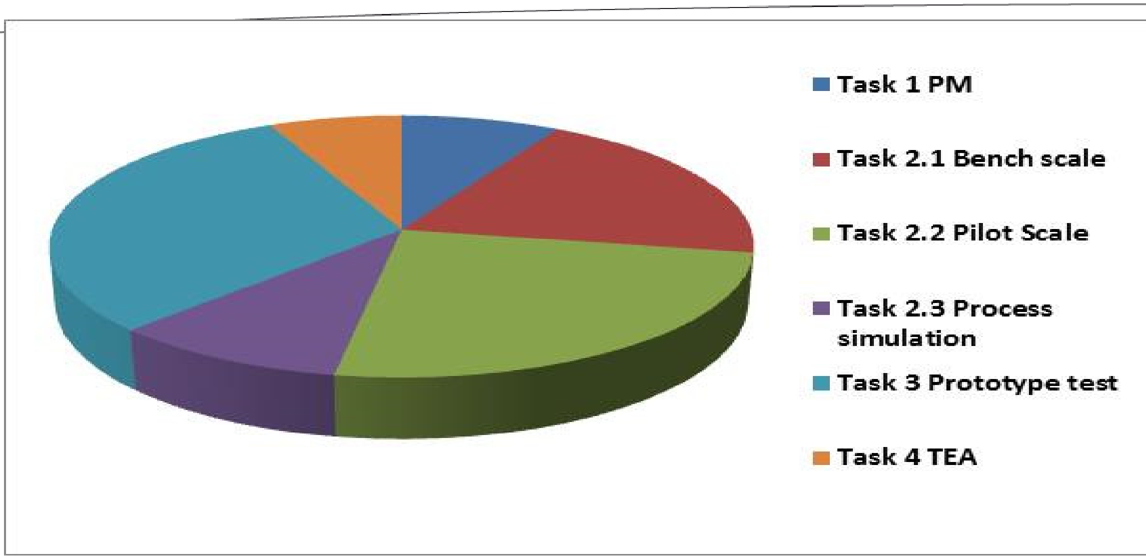
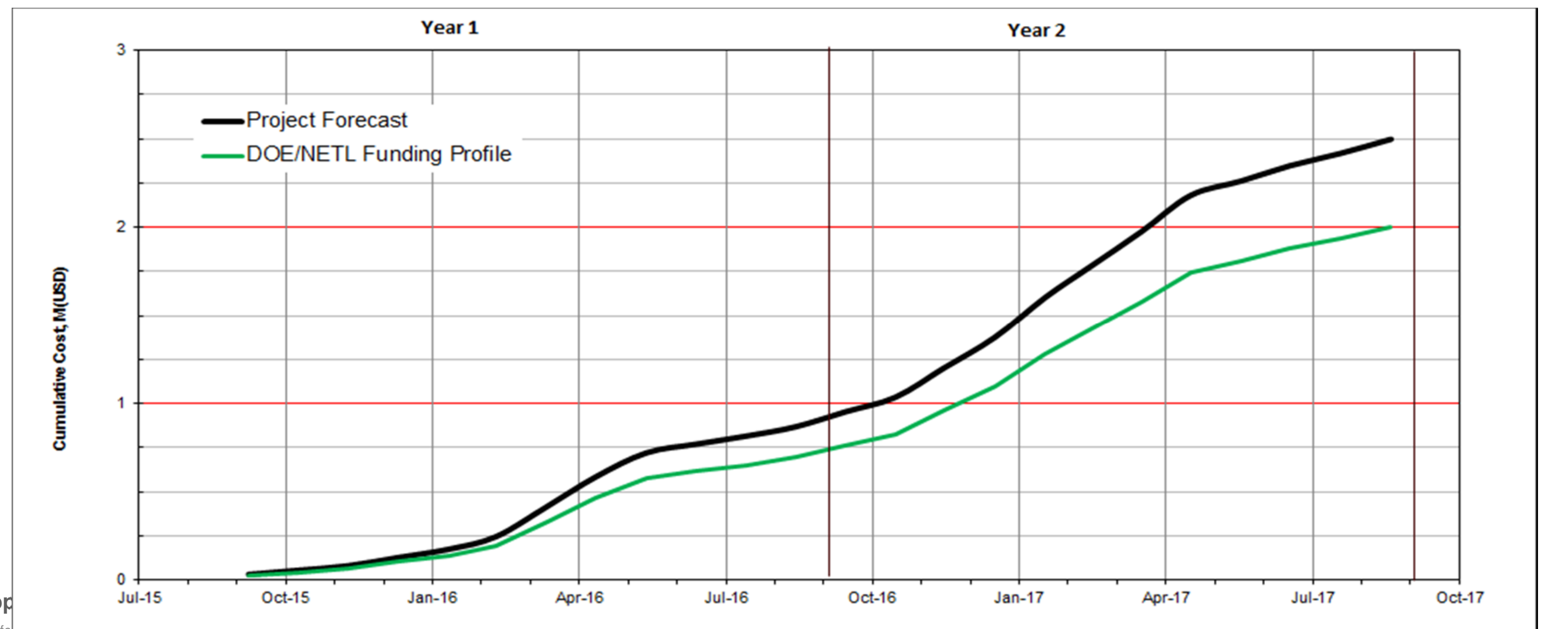
FE25073: Budget and spend rate





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Project Management Plan including Risk Management

- The PMP for FE25073 has already been submitted to Program Manager (Briggs White)
- Alstom's R&D project execution process will be used to clearly state objectives, define work packages, and corresponding WBS, scope, schedule, and budget
- Project/Risk management includes:
  - Weekly review of the inception-to-date budget and schedule.
  - Monthly, quarterly, and yearly progress reports are required by Alstom management (consistent DOE requirements)
  - The Task list is used to define the work to be accomplished.
  - The Milestone log will indicate progress on the main program activities.
  - The Project Timeline will be used to compare actual schedule performance to the predicted schedule.
  - The Project costing schedule will be used to compare project actual costs versus predicted costs.
  - DOE review at the end of the project is used to determine if the project has met all of the project milestones.

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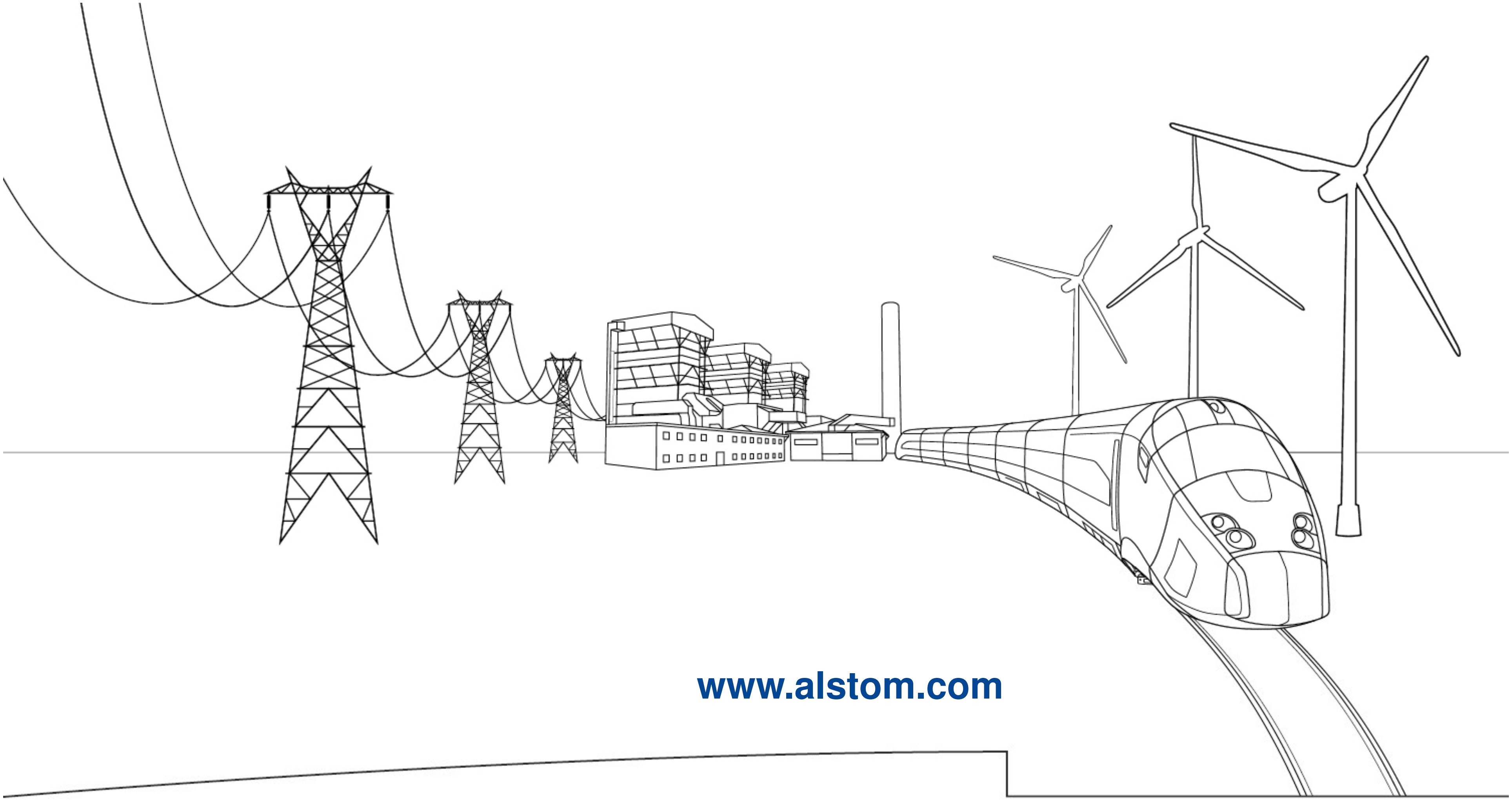
Project Risks

Risk Category	Risk Description	Probability (Low/Med/High)	Impact (Low/Med/High)	Overall (Low/Med/High)	Risk Mitigation Strategy
PROJECT RISKS					
Financial	• Funding reduction • Initial funding not adequate to meet deliverables because of change in activities based on work just completed	medium	medium	medium	• Monitor progress and budget on a timely basis using proven Alstom procedures and best practices • Ensure design and material selections are robust to minimize breakdown and repair • Adjust scope to initially include and address high priority technology items by maximizing the use of the UND and Alstom's 100 mm FBC and PSTF facilites to stay within budget • Alstom may increase its own funding if it is deem important to complete priority item
Known Technical Gaps	See also Technical Gap Table Phase II proposal • Achieving low or no oxygen demand of product gas at process terminal point • Solids Oxygen Carrier enhancement • Sorbent Reactivation • Improving Coal Volatile cracking • Sulfur Capture / Loss – Reducer, Oxidizer, Solids Inlets • Process Control (Steam side) • Sulfur Sorbent enhancement	medium	high	medium	• Determine product gas specification leaving reducer prior to improvements under this program • Determine best oxygen carrier combination through extensive bench tests using TGA, DTSF, 100 FBC and 100 PSTF. • Test and fine tune controls/operating parameters across load range • Test and optimize coal & limestone type/size to give acceptable performance • Test and determine performance of sorbent enhancement materials • Determine if the augmentation of another solids oxygen carrier is required.
Future Technical Gaps	Risks not addressed or found after the completion of all LCL programs	Medium	Medium	medium	Discover, test and mitigate risk during current LCL programs



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