Innovations in Oxidation Technology to Enable Market Growth

Composites World, 2012: Carbon Fiber,
Wednesday, December 5, La Jolla, CA

Bill Stry, PhD, Senior Process Technology Engineer

Agenda

- Introductions
- State of The Industry
- Challenges
  - High growth potential requires more efficient and cost effective carbonizing lines
    - Focus on oxidation ovens as this part of the process is the highest energy consumer
  - Anticipating the needs of alternate precursor materials

Introduction: About Harper

About Harper

Core Skills:

- Scale up of New or Challenging Processes
  - 200°C – 3000°C
  - Atmospherically Controlled
  - Continuous Processing
- Construction Techniques in Metallic > Ceramic > Graphitic
- Integrated Systems Design – Plant Supply
- Complex Flows of Advanced Materials
- Precise Control of Gas - Solid Interactions

About Harper

Advanced Thermal Systems for Fiber Processing

- PAN based C-fiber
- Pitch based C-fiber
- Rayon based C-fiber
- Alternative Precursor Development
- Carbon Nano Tubes / Fibers
- Carbon Fiber Recycling

A Broad Experience Base in a Range of Carbon Processes

State of the Industry

State of the Industry: Review of Scales of Operations

Scales	Size Range (mm Width)	Capacity
Production	1000mm-4200mm	100 tpy to more than 4000 tpy
Industrial Scale Pilot	300mm-1000mm	20 tpy -100 tpy
Micro Scale (University, Institute)	<100mm	Less than 3 tpy

State of the Industry: Review of Scales of Operations

- Modern Line Speeds

  10 – 15 m/min for a state of the art line

- Oxidation Oven Capacities

  500 – 750 kg/hr feed rate of PAN

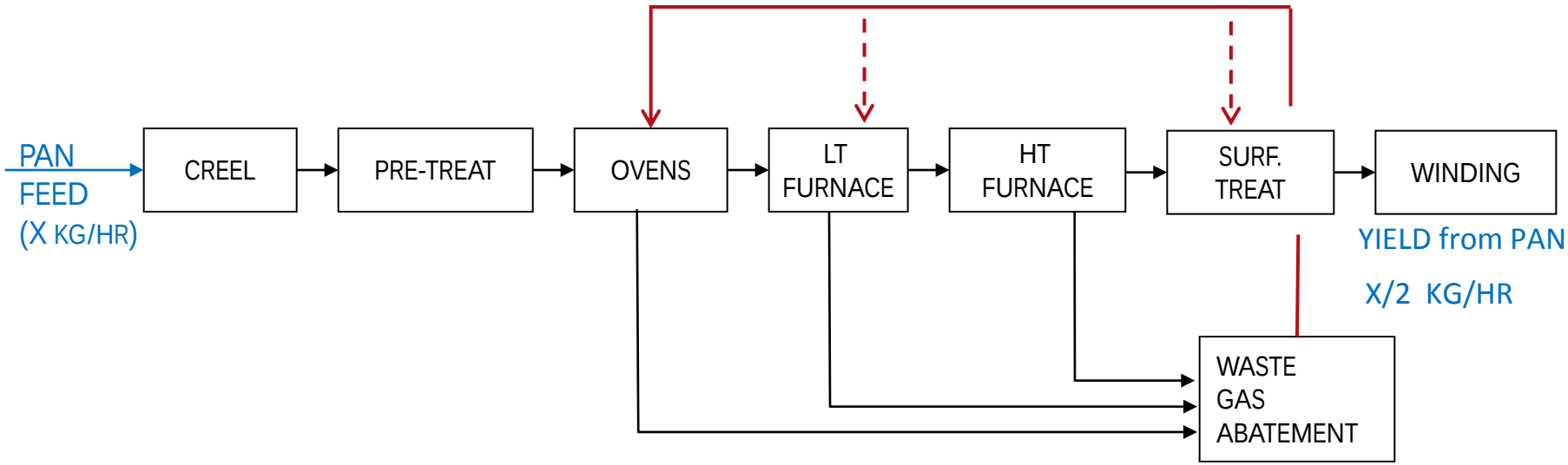
  500 – 1000 m Overall Heated Length

  Typically 6 – 8 Temperature Zones

  Widths up to 4m -- 3m wide designs are becoming more common

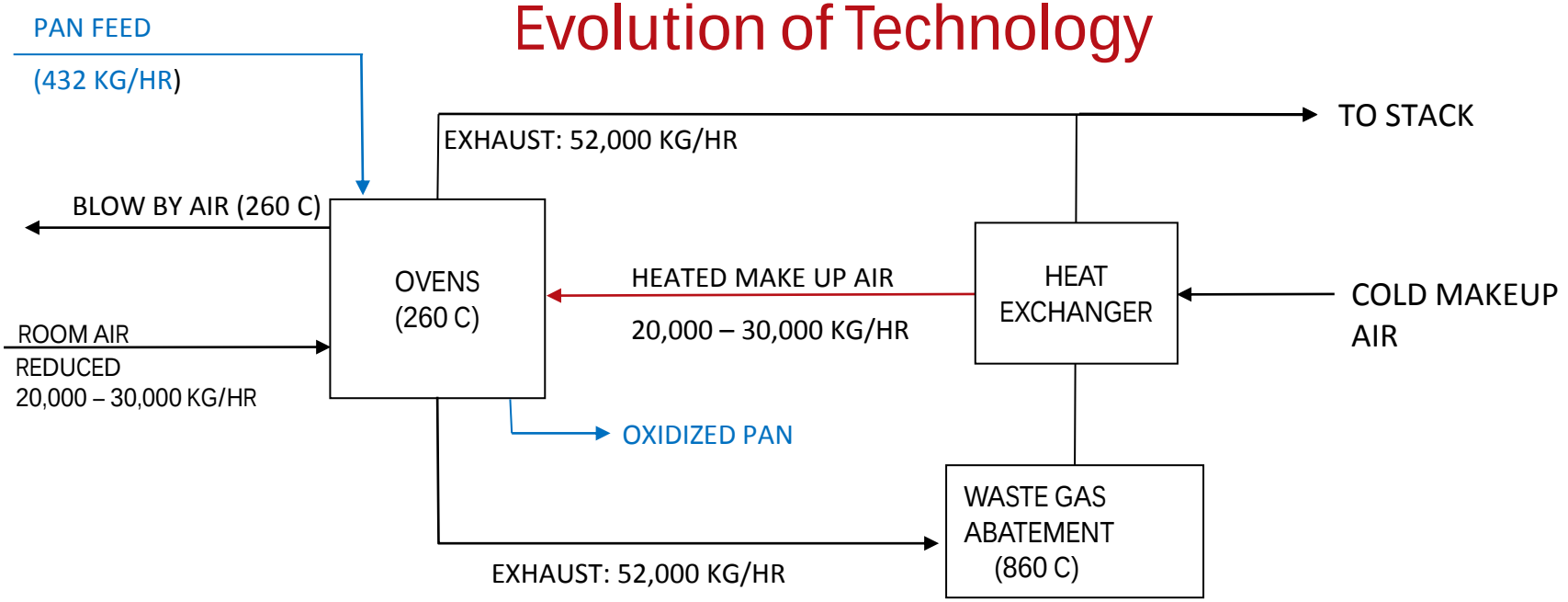
  Unsupported Heated Lengths typically up to 15m

State of the Industry:
Carbon Fiber Conversion Process



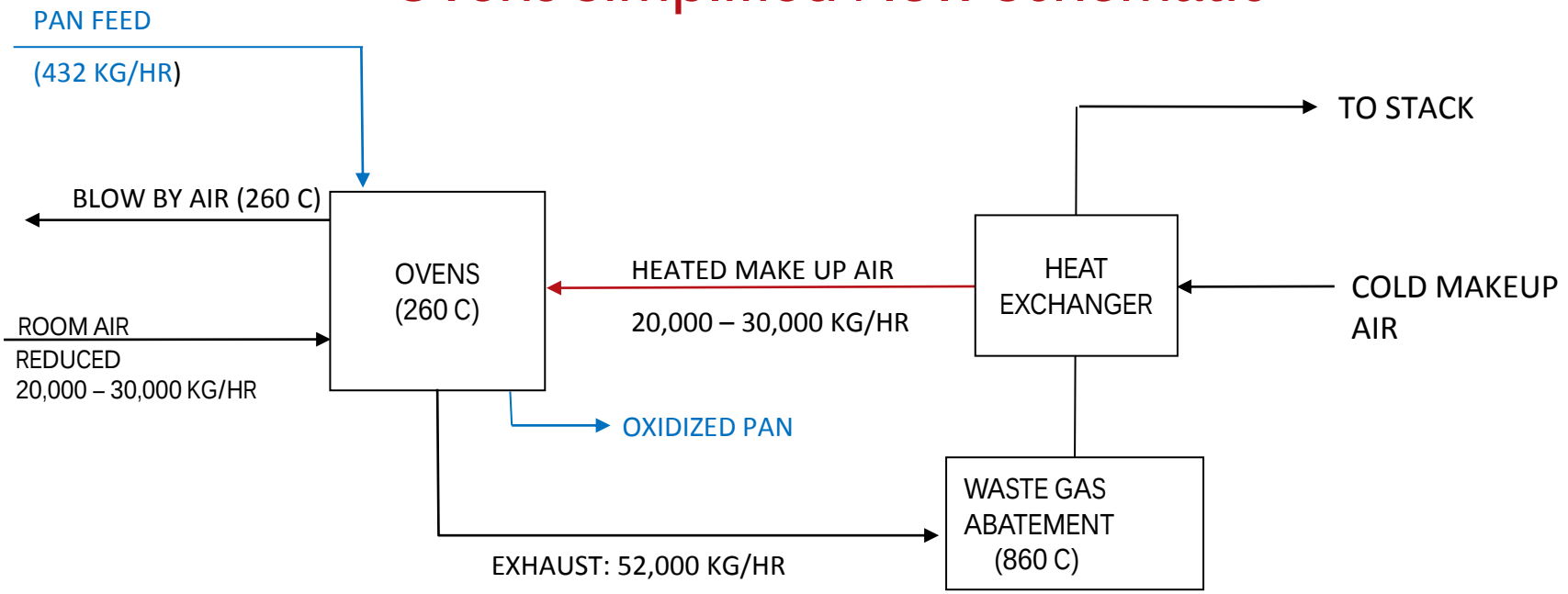
- Complexity and Cost added through Waste Gas Treatment
- Opportunity for Energy Recovery and Cost Reduction
- Opportunity for increased yield (via Alternate Precursors)

1500 Ton/Year Line - Simplified Oven Flow Schematic Evolution of Technology



- Originally, Oven Systems Operated without Waste Gas Abatement – Some Still Do
- Addition of Waste Gas Abatement Reduces Emissions but Increases Cost
- Use of Recovered Energy at Ovens Lowers Operating Cost

State of the Industry: 1500 Ton/Year Line - Ovens Simplified Flow Schematic



- Room Air infiltration (typically at lower portion of slots) reduces the effectiveness of heat recovery
- Significant Opportunity for Energy Recovery and Cost Reduction through reducing Room Air infiltration and Blow By Air

Treatment of Oxidation Oven Exhaust:

Integration & Heat Recovery
2m and 3m (6 Zone Designs)

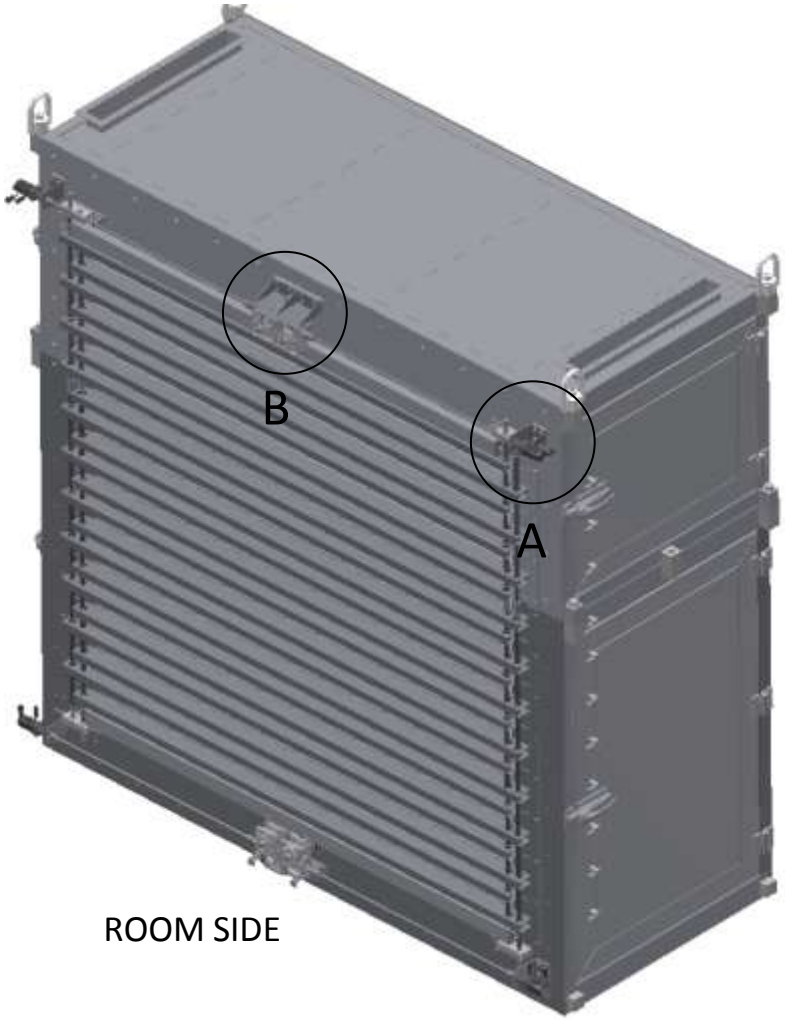
Line Width	m	2m Wide	3m Wide
Oxidation Oven Zones	#	6	6
Exhaust Rate	Nm3/hr	20760	43200
Exhaust Temp	C	260	260
Ambient Temp	C	25	25
Energy Lost (with no heated makeup air)	kw	1660	3454
Energy Lost (with 50% heated makeup air)	kw	830	1727

Challenges

- Increased energy efficiency through reduction of cold make-up air infiltration
- Increased CF yield through alternate precursors

Harper 3M Oxidation Oven

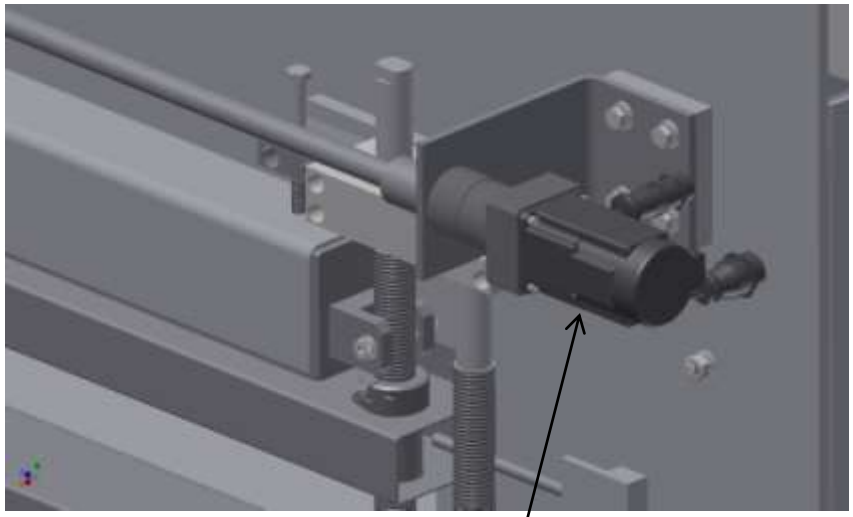
Harper 3M Oven End Seal



ROOM SIDE

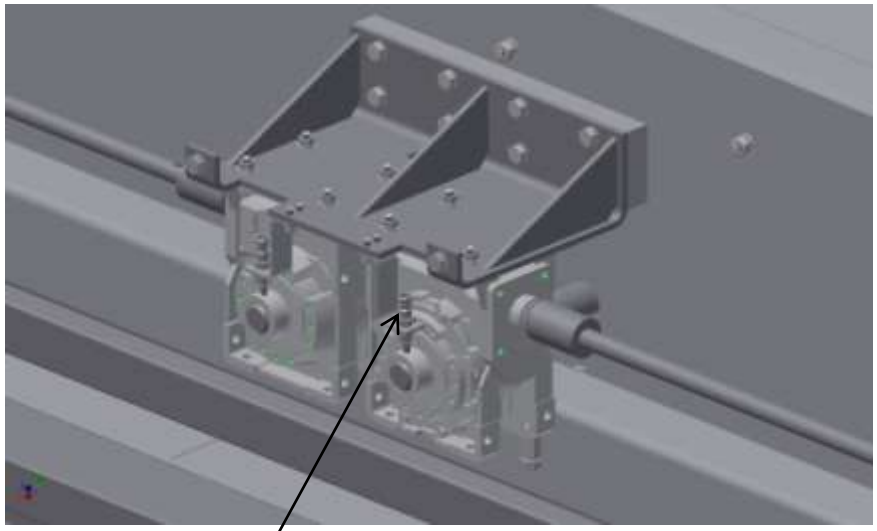
OVEN SIDE

Harper End-Seal Details



A

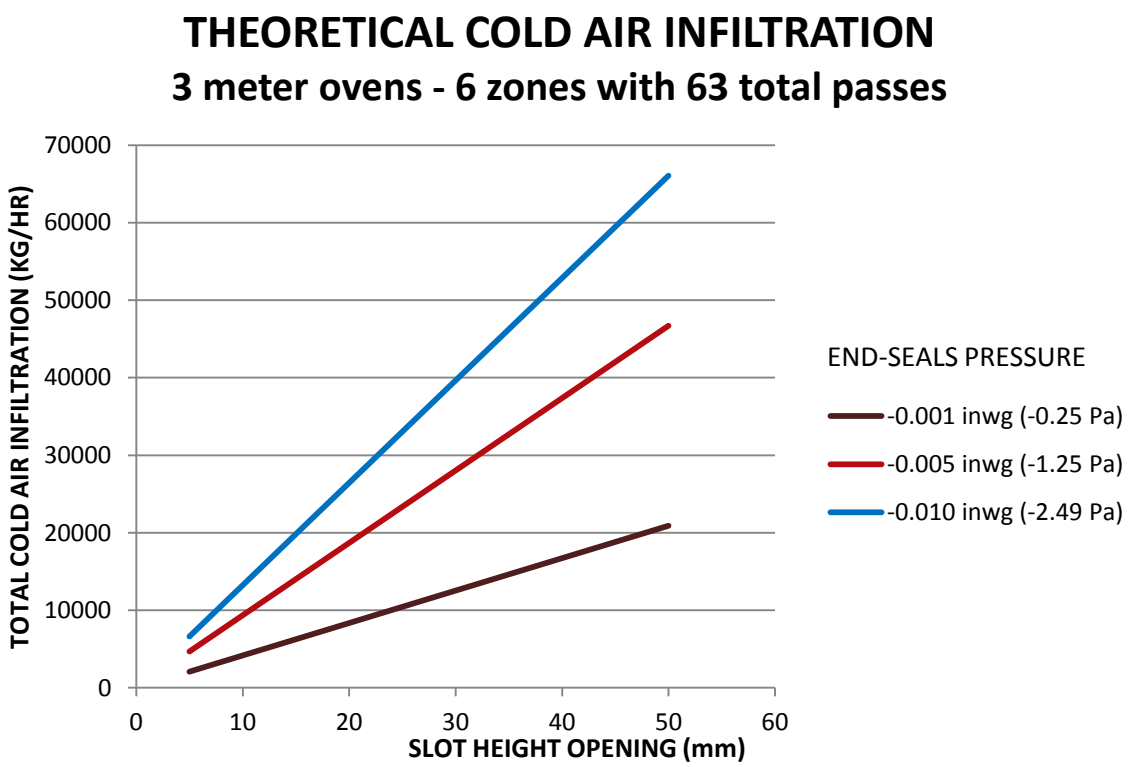
MOTORIZED CONTROL OF SLOT OPENINGS



B

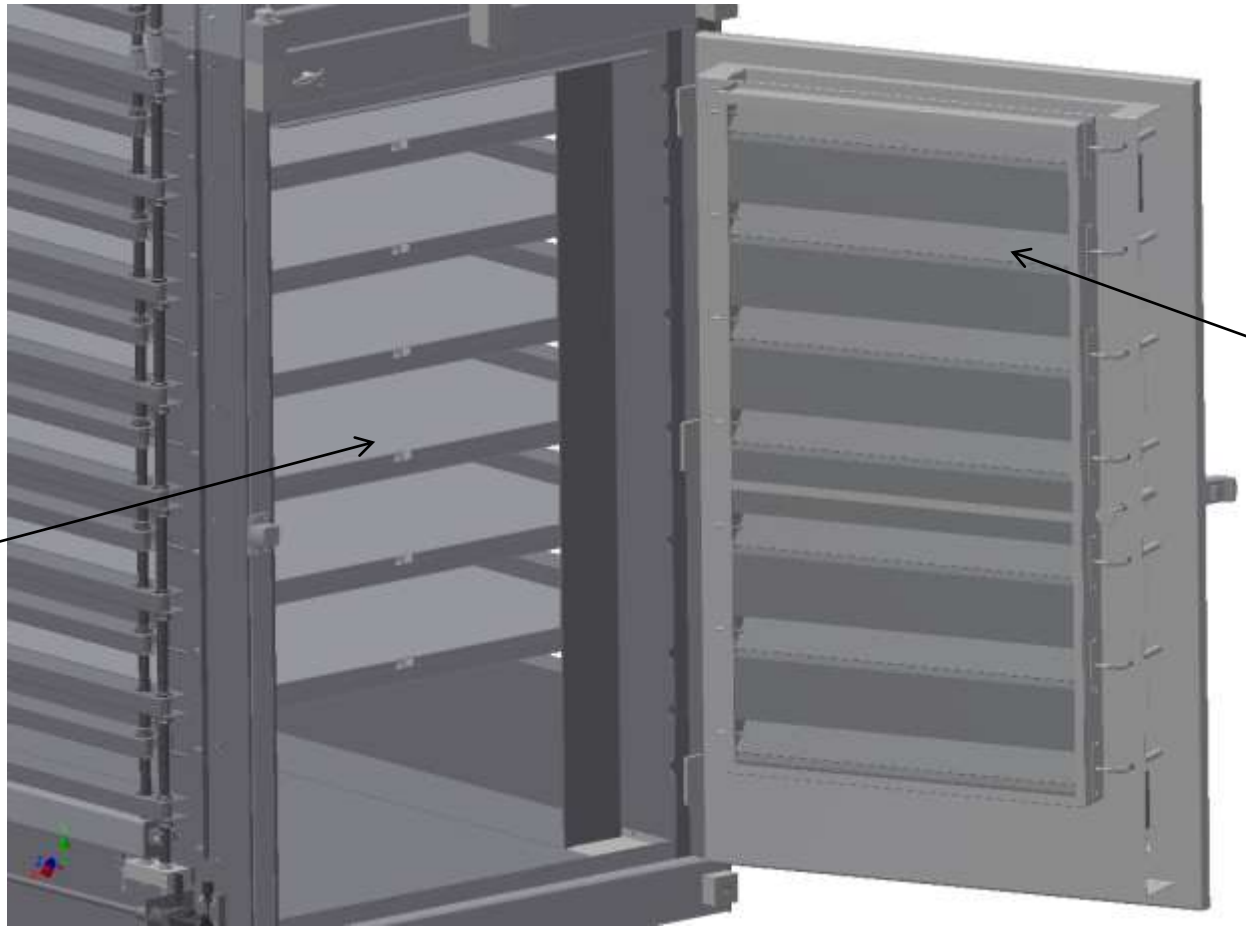
PERMANENT KNOWN ZERO POSITION

Better Slot Position Control Means Better Control of Air Infiltration



In practice, the Chimney Effect creates non-uniform pressure – negative at the bottom; positive at the top

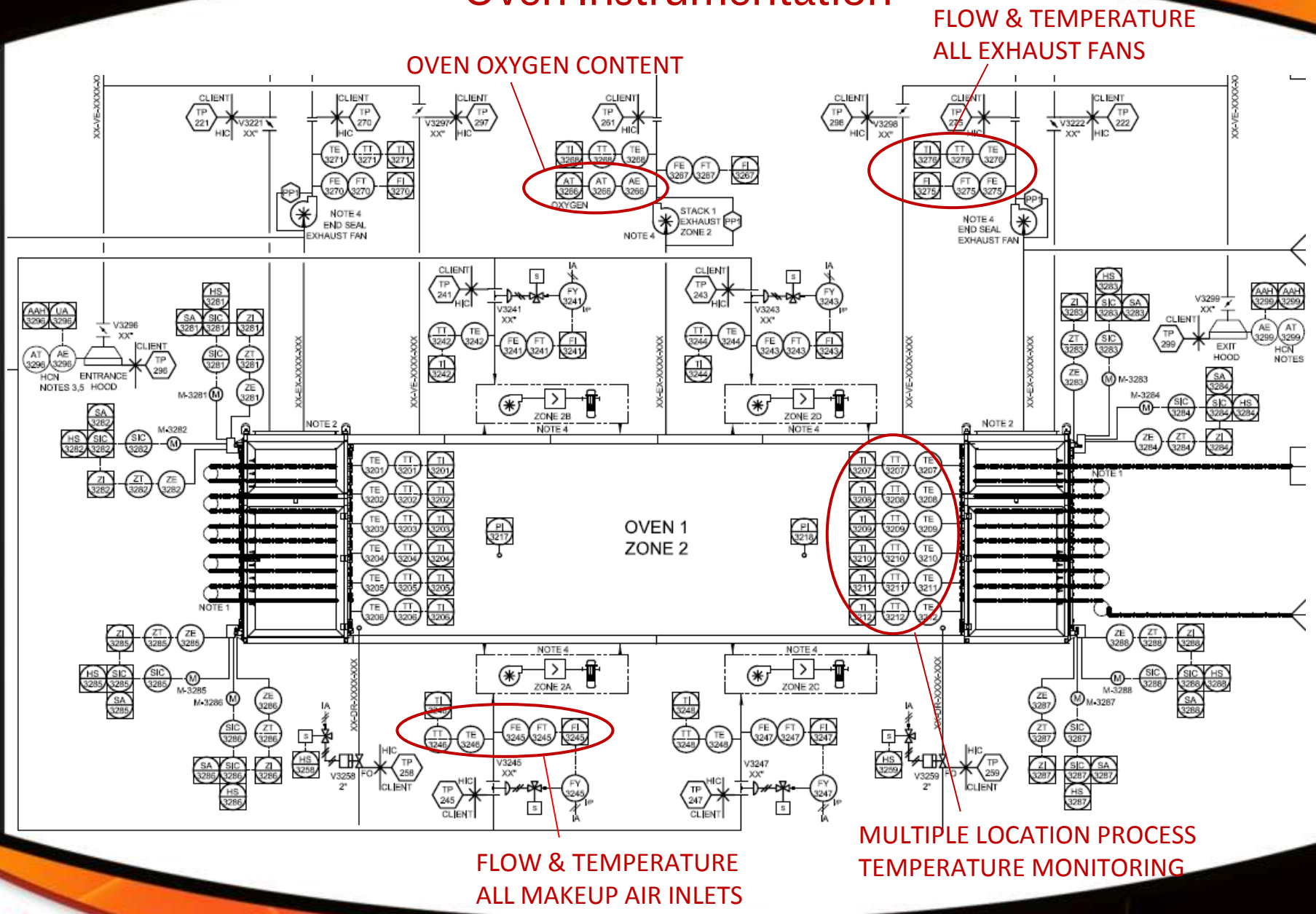
Harper End Seal Pressure Control



SEPARATION PLATES AT EACH TOW HEIGHT

EXTERNALLY ADJUSTABLE EXHAUST FLOW DAMPERS AT EACH TOW HEIGHT

Oven Instrumentation



Oven Instrumentation Benefits

- Measured flow rates at all exhausts and makeup air locations provides real-time knowledge of:
  - Rate of cold air infiltration (via conservation of mass)
  - Total flow and temperature of oven exhaust (inlet to abatement system)
- Measured oven oxygen content provides critical information for setting exhaust flow rates
- The combination of airflow rates, oxygen content, and slot height data enables a new degree of process optimization

Harper 3M Oven End Seal

PERFORMANCE GUARANTEE:

- Less than 25% of oven makeup air will be cold air infiltration

Harper Air Knife Advancements

- Enables introduction of hot makeup air from outside the slots
- Decreased risk of process contamination
- Reduced HCN present in the pass-back rolls area

Developed and Demonstrated through Empirical Research

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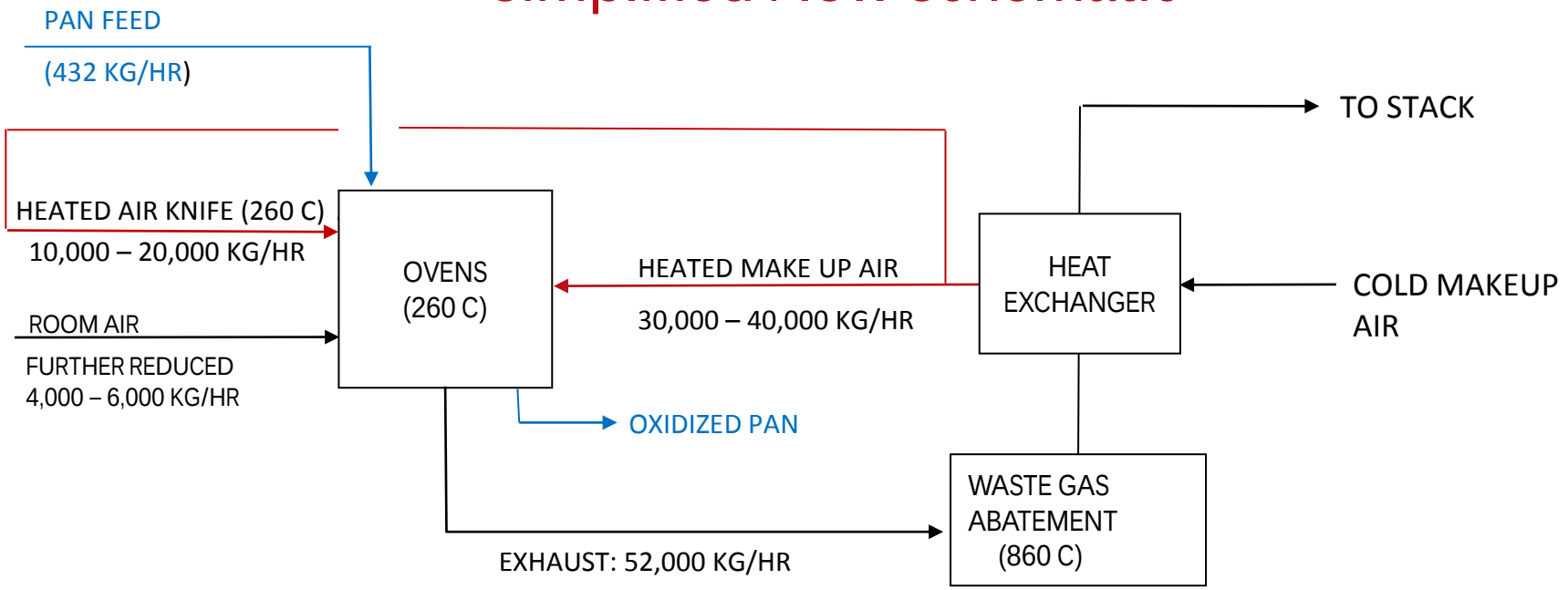
Harper 3M Ovens - Simplified Flow Schematic



- With Harper End-Seals Louver Position Control total air infiltration at the slots is reduced
- With Harper Air Knifes most slot infiltration is Heated Make-up Air, further reducing energy consumption
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Integration & Heat Recovery
2m and 3m (6 Zone Designs)

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Energy Lost (with 90% heated makeup air)	kw	166	346

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Hours per year		7000	7000
USD$ / kw-hr		$0.1	$0.1
USD$ / year		$1,040,000	$2,017,000

Challenges to Future Growth: The Carbon Fiber Yield Ratio

CF Precursors & Yields

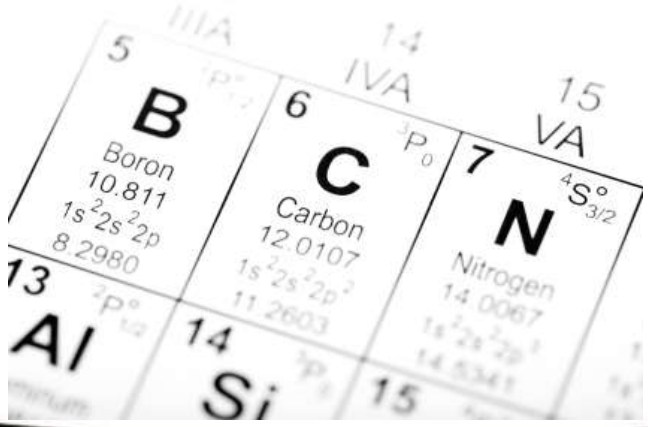
Chemistry	Maximum C- Recovery	Typical C-Recovery
▪PAN	68% max	(50% typical)
▪Cellulose	44% max	(20% - 30% typical)
▪Lignin	67% max	(typical ?)
▪Pitch	85% max	(w/o solvent)
		(25% - ?? w/ solvent)
▪Polyethylene	85% Max	(typical ?)
▪Polypropylene	85% Max	(typical ?)

The Precursor can have a Significant Impact on Yield and Carbon Fiber Cost

Processing Challenges for Alternative Precursors

Establishing Atmospheric Control in Oxidation will be required for Alternative Precursors and Alternative Processing Technologies

1. Chemical Control of Oxidation Atmosphere
2. Containment of Organic and / or Corrosive Off Gassing Species
3. Further Optimization of Heat Recover
4. Reduction of Wasteful Energy Consumption through Excessive Exhausting of Off Gasses



Harper Oven Features for Alternate Precursors

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- Belt transport for matted or felted product formats
- Driven pass-back rollers for slip prevention at low loading
- Internals with a high degree of corrosion resistance
- 3 Discreet airflow directions

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Summary of Advantages
Harper Oxidation Oven Technology

Establishing Atmospheric Control in Oxidation will be required for Alternative Precursors and Alternative Processing Technologies

1. Maximized Reuse of Energy Available from Waste Gas Abatement – Approaching the Target of Full Offset of Oven Heating Requirements
2. Instrumentation and Control Advances Allowing for a High Degree of Process Optimization
3. End-Seals that for Maximize of Effective Heated Volume and Drastically Reduce of Ingress of Room Air and the Chimney Effect

Cumulatively, the Benefits of Harper's Oxidation Oven Advances Provide Dramatic Reduction of Energy Required for Oxidation

Two to Three Fold Reduction In Consumed Energy

Thank you for your time!

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