CRITICAL OXIDATION OVEN DESIGNS TO ENABLE RESEARCH SYSTEM GOALS

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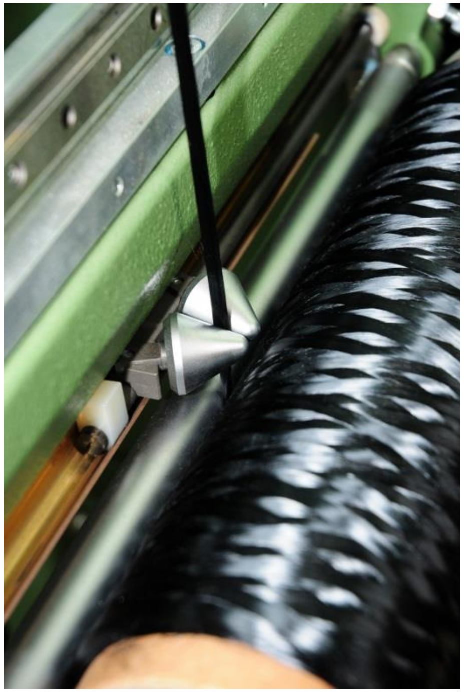
Introduction

- What is critical in the CF oxidation process?
  - Heat transfer
  - Atmosphere composition
  - Oven sealing
- All of these must be managed as processes go from research scales to pilot scales to commercial production



Carbon Fiber Carbonization Process – Scales of Operation

Scale	Size Range (Tow-Band Width)	Capacity
Commercial Production Line	1000 – 4200 mm	500 - 4000 ton/year
Pilot Line	300 -1000 mm	20 - 100 ton/year
Microline	≤100 mm	Less than 10 ton/year
Scientific Line	Fractional tows (<1k or less than 1,000 filaments)	Less than 1 ton/year

Courtesy of Georgia Institute of Technology

Courtesy of Oak Ridge National Laboratory

Oven Heat Transfer

- Convection air flow both heats and cools the fiber material
- For exothermic material at steady-state

$$Dm\ DH = h\ A\ (T_{Product} - T_{Oven})$$

where

Dm is the mass rate of fiber , DH is the enthalpy of reaction,

h is the heat transfer coefficient, A the heat transfer area, and

$(T_{Product} - T_{Oven})$ is the elevation of the fiber temperature above the oven temperature

Oven Heat Transfer

- The heat transfer coefficient depends primarily on air velocity

- Textbook correlations can be applied

$$Nu_D = Re_D \, Pr^{1/3} \, (f/8)$$

Rearranging the above equation….

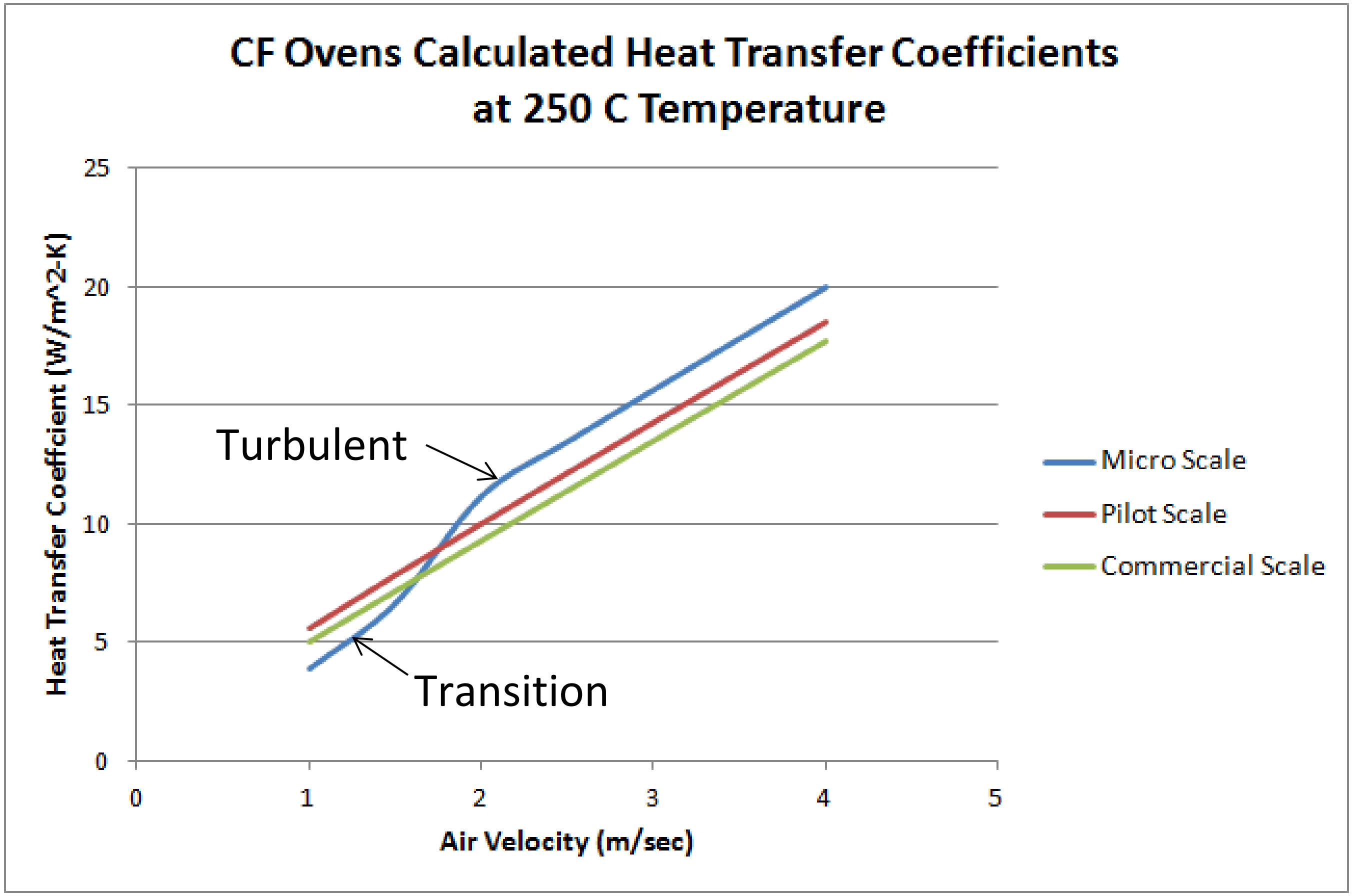
$$h = (Pr^{1/3} \, k/n) \, (f/8) \, V$$

Where Pr, k, and n are air properties

f is the friction factor, and V is the air velocity

Oven Heat Transfer

- The previous equation can be applied to different sizes of ovens



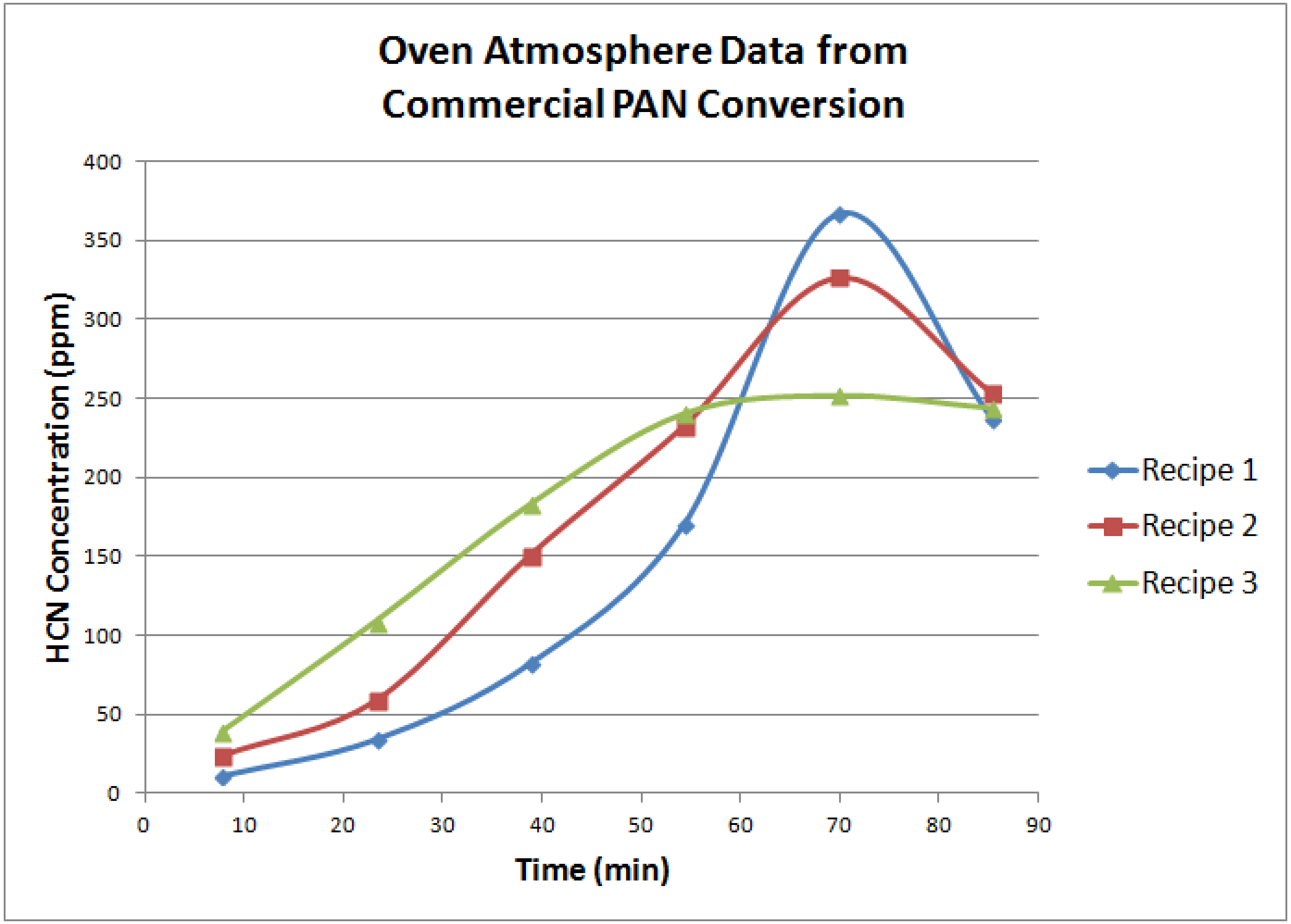
Oven Atmosphere Composition

- Stabilization reactions will produce off-gas components
  - For PAN, most important component is HCN

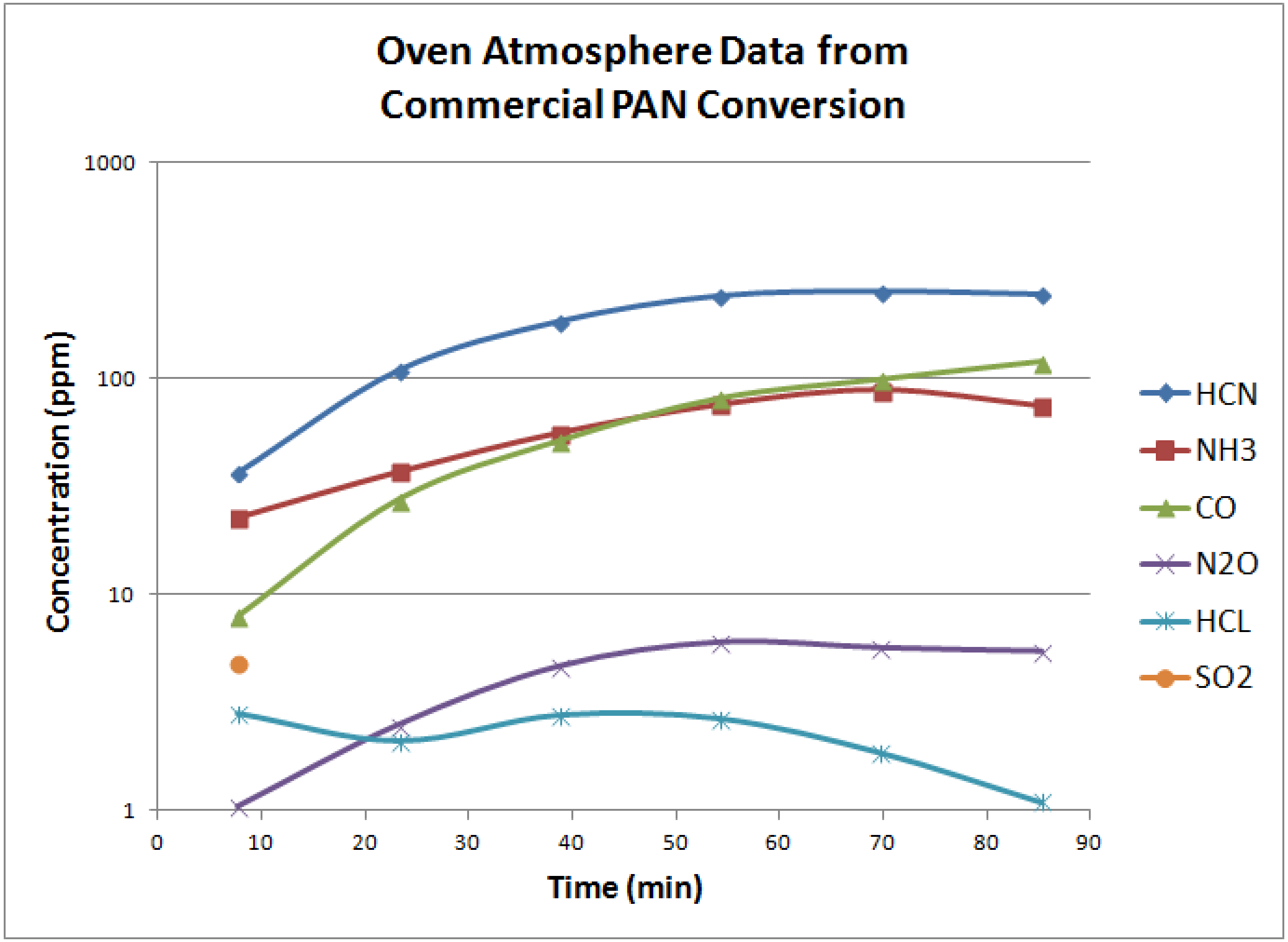
    IDLH = 50 ppm; NIOSH REL = 4.7 ppm

    - Also Cl, S, and Si known bad actors
  - Alternative precursors may have these or other components

Oven Composition Example



Oven Composition Example



Oven Atmosphere Composition

- Concentrations increase with higher conversion rate, and concentrations decrease with higher oven exhaust rate
- The higher the concentration inside the oven, the higher the concentration in the workspace outside the oven
- Knowledge of the critical component compositions is valuable
  - For health and safety
  - For product quality (final CF properties)
  - For energy efficiency (optimization of exhaust rates)

Measurement of Oven Composition

- *Need access to the oven atmosphere*
    - *At least at 2 locations per zone*
    - *Even better, at each pass*
- Instrumentation (FTIR, GC, Mass-Spec, Stack gas analyzers)

Harper's portable FTIR unit

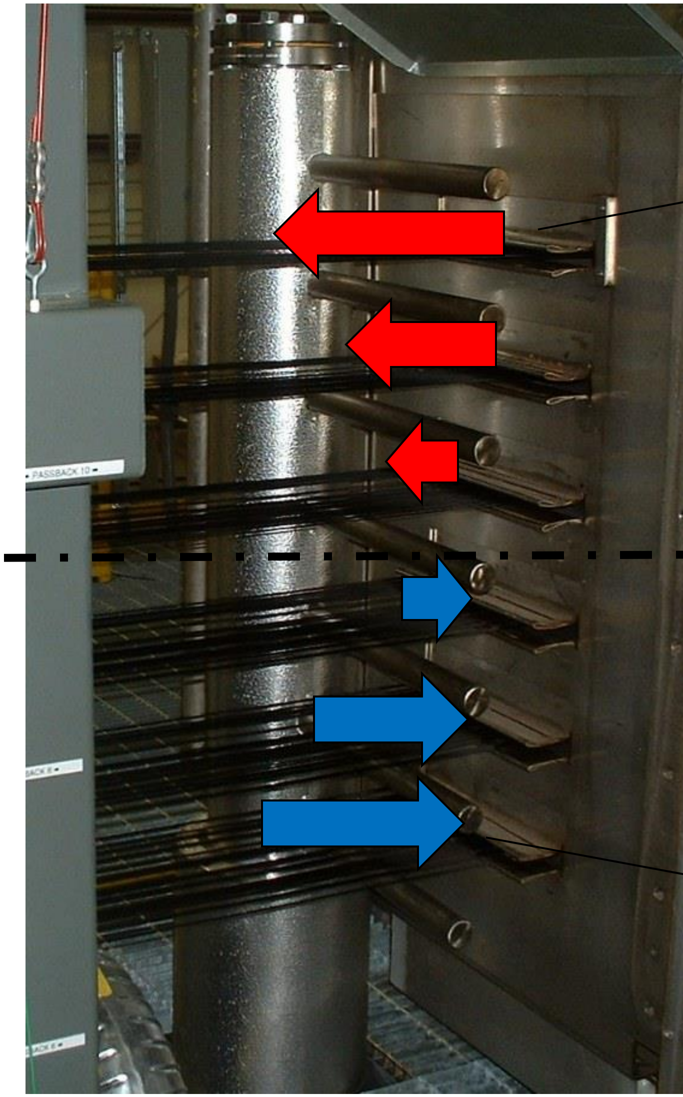
Oven Atmosphere Sealing

- Oven sealing affects the operation in several ways
  - Health and Safety
    - Escape of oven gas means exposure to HCN and other off-gases
    - Escape of oven gas creates a hotter workspace
  - Efficiency
    - Ingress of ambient air cools the oven ends reducing the useful volume
    - Ingress of ambient air adds to waste gas abatement cost

Oven Atmosphere Sealing

- *Buoyancy drives the problem*
- *Oven height creates buoyancy*

Neutral pressure height

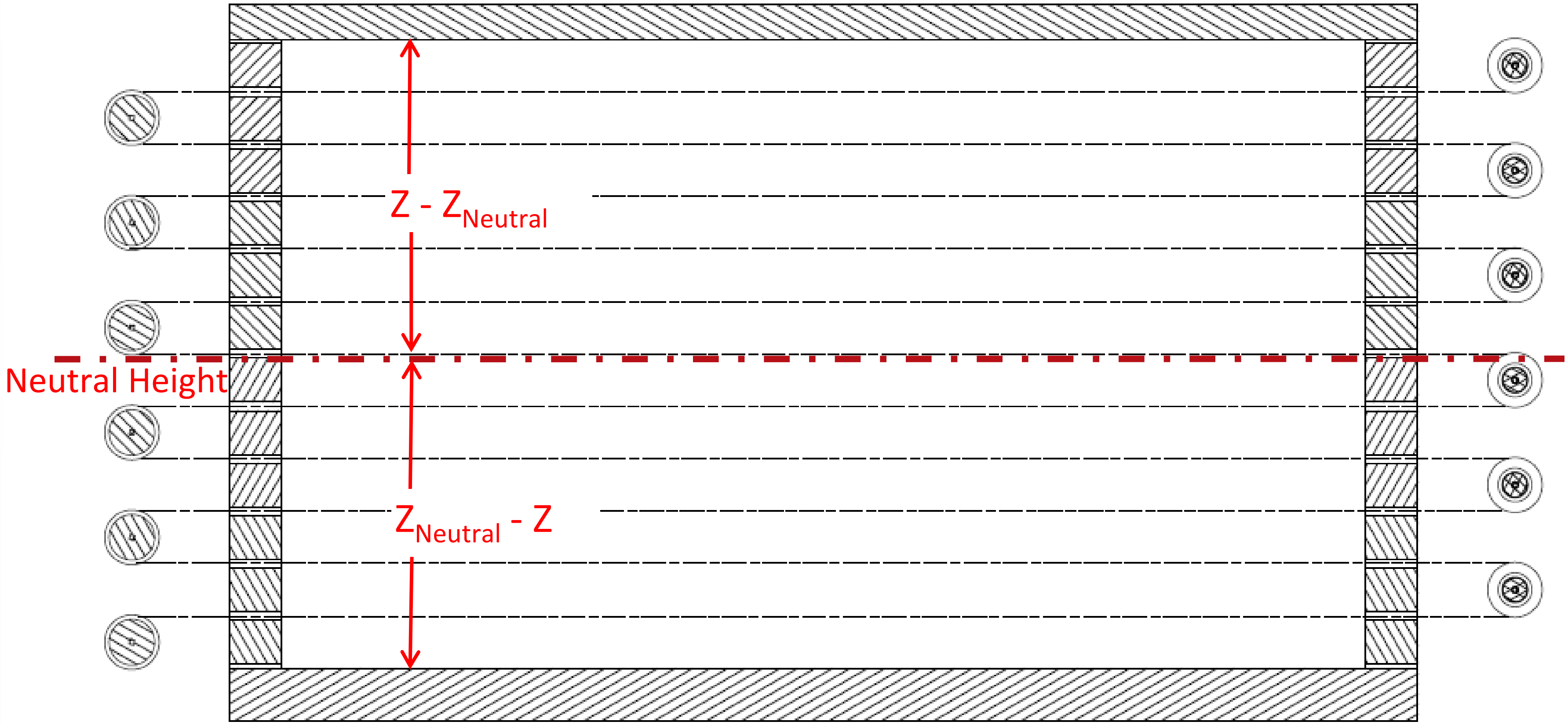


Oven gas escapes at upper slots

Cold air infiltration at lower slots

- *This phenomenon is called the chimney effect*

Oven Atmosphere Sealing



Oven cross-section schematic

Oven Atmosphere Sealing

- Pressure difference over the height of the oven is approximately:

$$\Delta P = \rho_{amb}\, g\, (Z - Z_{neutral})\,(1 - T_{amb} / T_{oven})$$

- Flow through the oven slots follows from Bernoulli principle:

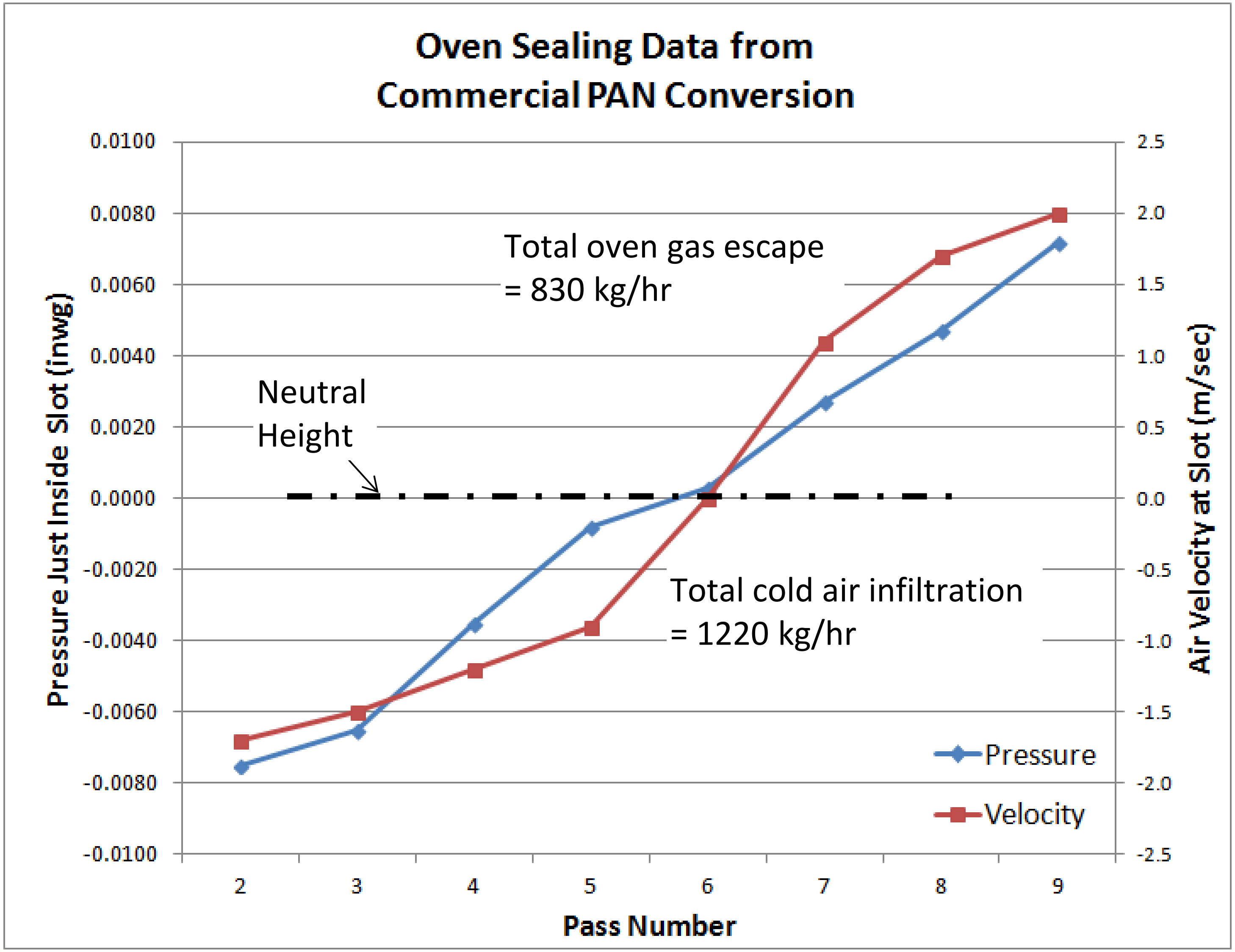
$$\Delta P = K\, ½\, \rho V^2$$

- V is the air velocity at the slots, so that flow Q into or out of a slot is:

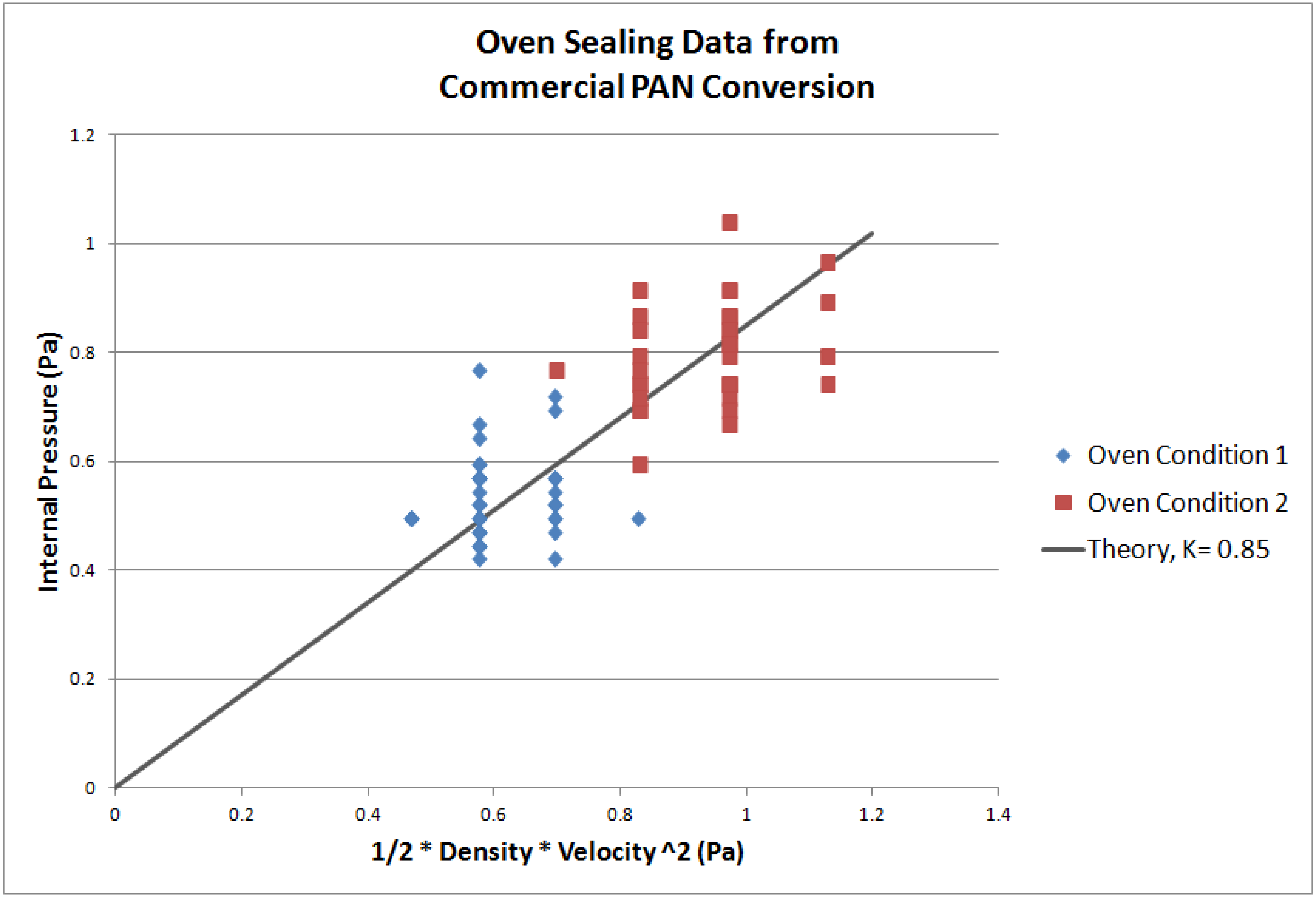
$$Q = VA = V(\text{Slot gap x Slot width})$$

Larger (higher) ovens have greater velocities and wider slots

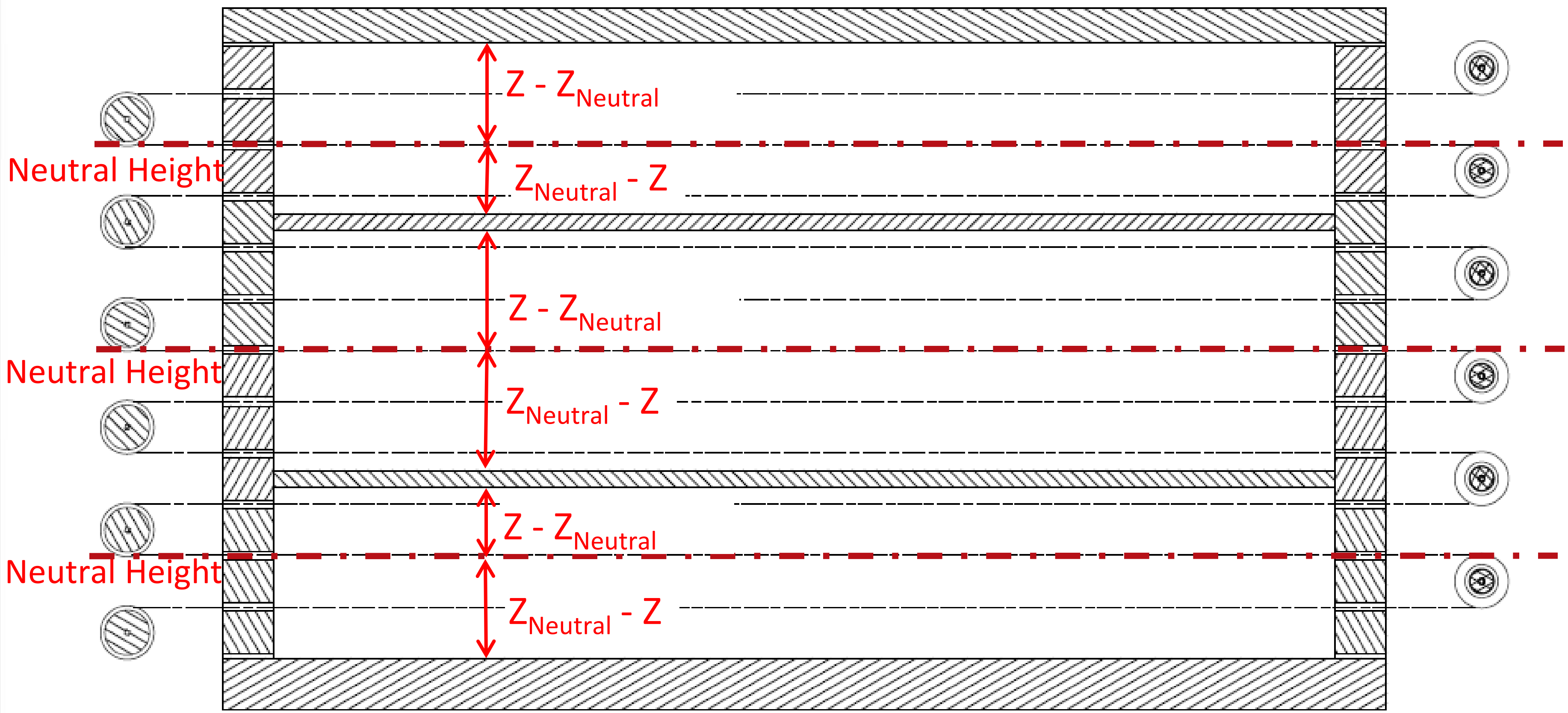
Oven Atmosphere Sealing



Oven Atmosphere Sealing



Oven Atmosphere Sealing



Oven cross-section schematic

Harper Micro-Line Oven - Flow Schematic

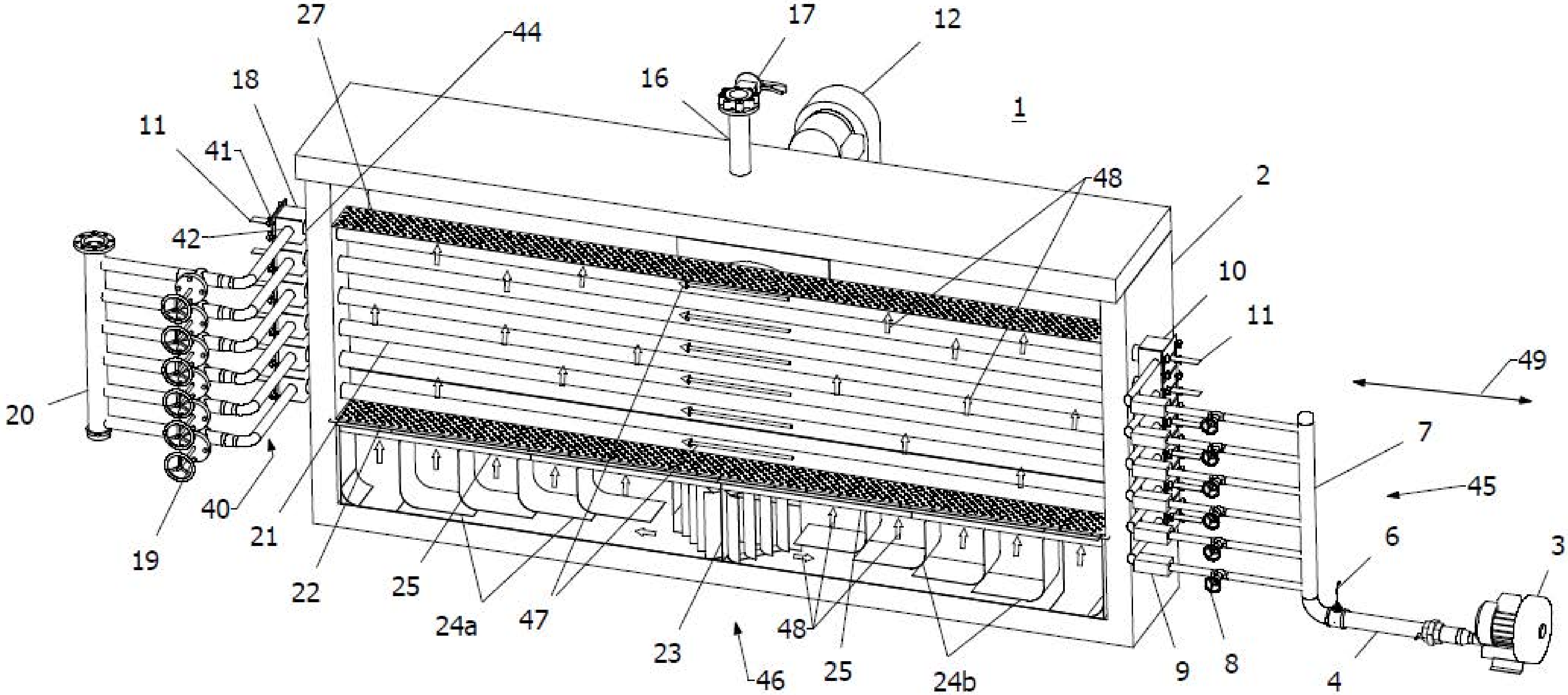


FIG. 3

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Summary / Key Points

- Heat transfer largely independent of oven scale
  - Match air velocity, match heat transfer
  - Pilot scale ovens can have low turbulence or laminar flow

- Reaction kinetics independent of oven scale
  - Match temperature and velocity, match kinetics
  - Off-gas composition useful indicator of reaction
- Oven sealing depends on scale of operation
  - The larger the oven, the larger the chimney effect and the harder to seal

Thank you for your time!

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