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PLACE

POLYMERS • LAMINATIONS • ADHESIVES • COATINGS • EXTRUSIONS

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Gel Troubleshooting

Presented by:
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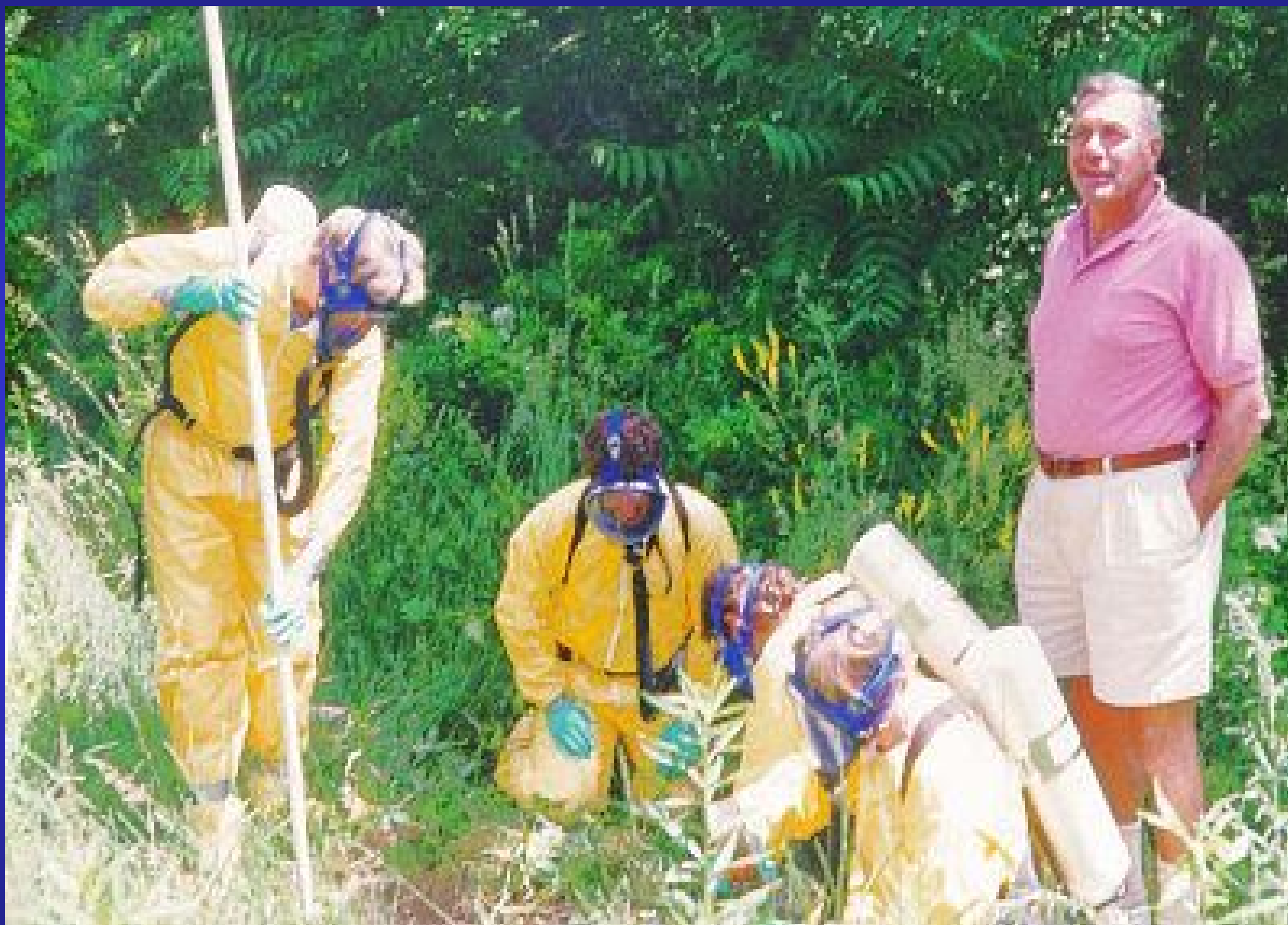
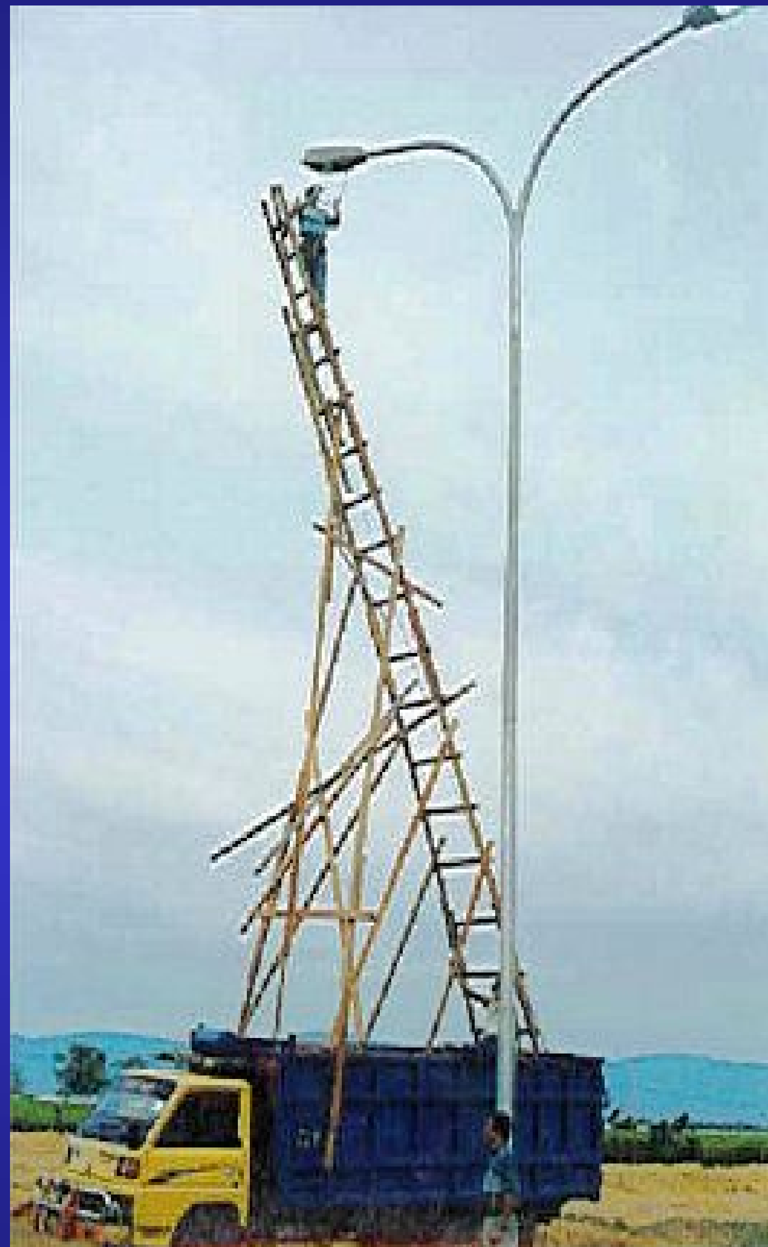
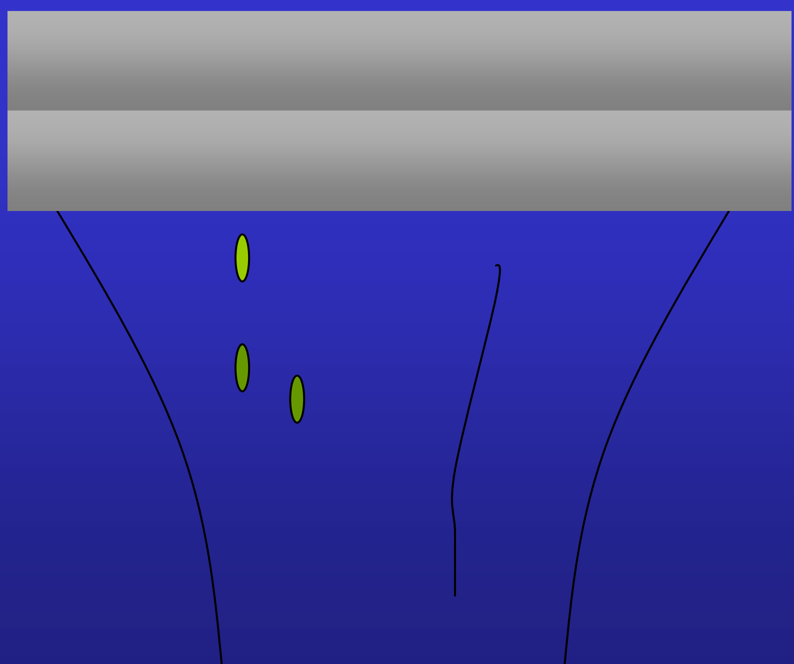
Davis-Standard, LLC

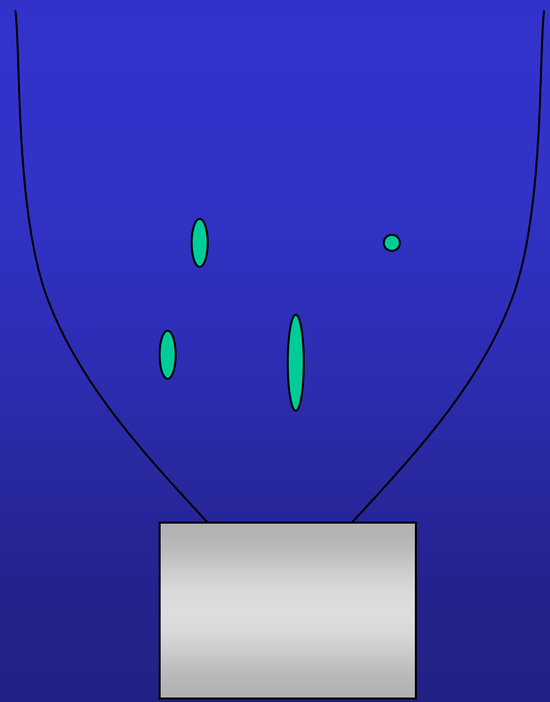




Definition of a Gel

- Film Imperfections or defects developed during forming due to disturbances in the polymer flow





Classification of Film Imperfections

- Contamination
- Un-melt
- Un-mix
- Thermal degradation

Origins
Gels can be created or introduced during

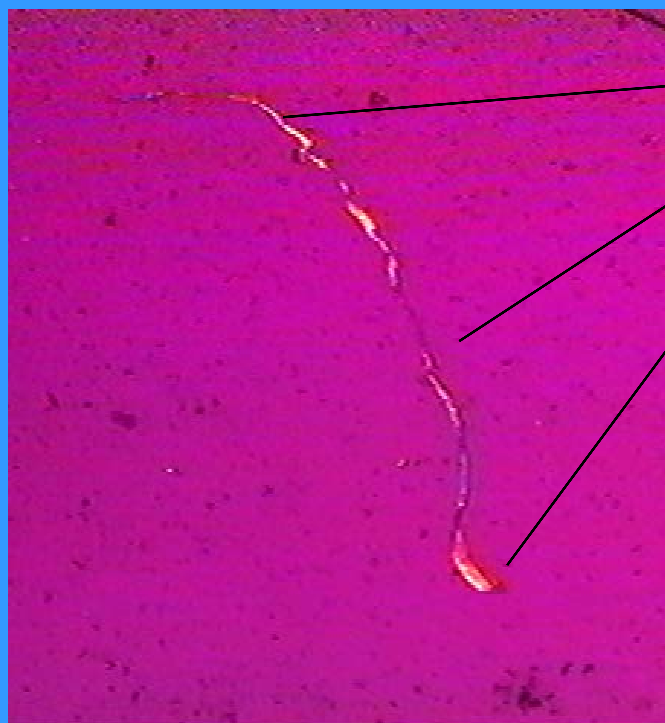
- Polymerization
- Equipment-Screw Design
- Fabrication

Contamination

Gauge Bands/Gels

- Contamination
  - Fiber

Fiber Gel After Melting



Gel

Polarized light (w/ 1/4 filter)

Gauge Bands/Gels

- Contamination
  - Fiber
  - Resin Contamination

Resin Contamination

LDPE

- Polyolefin
- More Crystalline
- Thermally stable
- Adhesion through oxidation
- 600°F - 630°F processing temperature

EVA

- Acid functional groups
- Less Crystalline
- Not thermally stable
- Adhesion through vinyl acetate groups
- 450°F processing temperature

Cross Contamination Solutions

- Better Housekeeping
- Better purging
- Work with resin companies

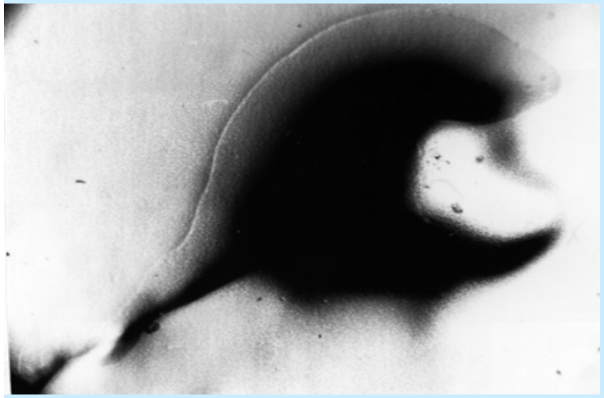
Gauge Bands/Gels

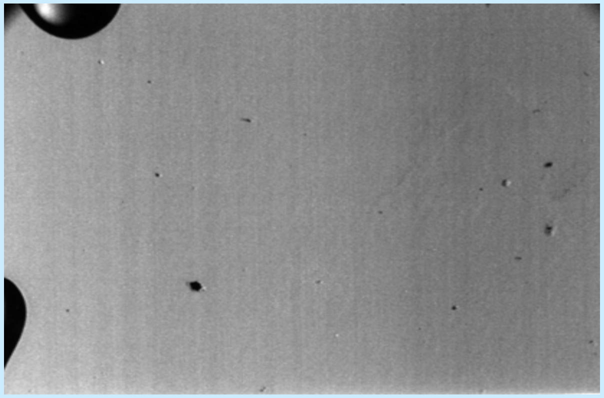
- Contamination
  - Fiber
  - Resin Contamination
- Un-melt and un-mix

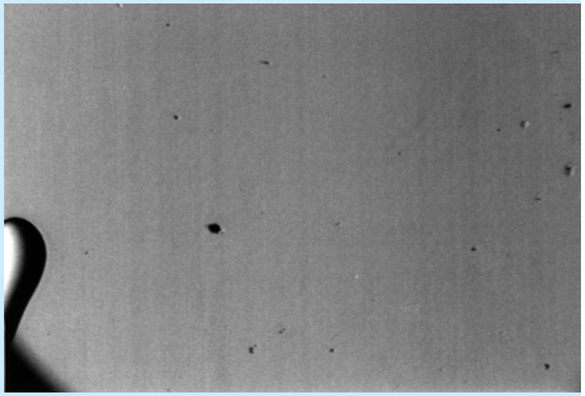
Hot Stage Microscopy

- Slowly heat sample to above melting point.
- Transmit light through cross-polarized filters.
- Observe melting point of film/gel and note any birefringence effects.

Unmixed Gel Characterization







50°C → **150°C** → **50°C**

Unmixed Gel

Gauge Bands/Gels

- Contamination
  - Fibers
  - Cross contamination w/other Polymers
- Un-melt & un-mixed
  - Bad Concentrate

Mixing
Master Batch Concentrates

- Compatibility of base resin
- quality of dispersive mixing
  - agglomerates

Gauge Bands/Gels

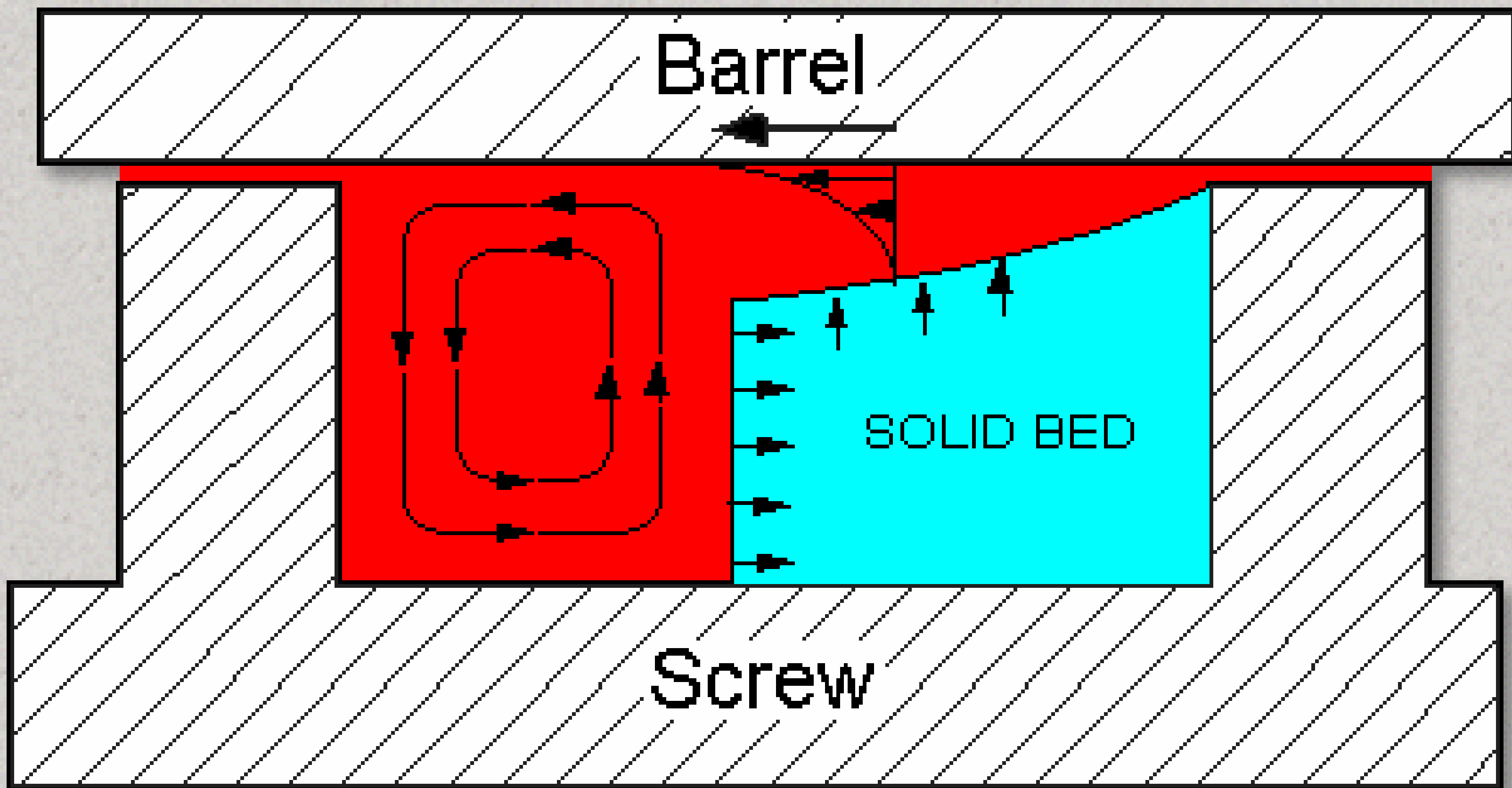
- Contamination
  - Fibers
  - Cross contamination w/other Polymers
- Un-melt & un-mixed
  - Bad Concentrate
  - Blends with different melting rate

Mixing
Polymer Blends and Additives

- Thermodynamic compatibility of components (conductivity)
- Large differences in melting points of blend components
- Large viscosity ratios between minor and major components

Gauge Bands/Gels

- Contamination
  - Fibers
  - Cross contamination w/other Polymers
- Un-melt & un-mixed
  - Bad Concentrate
  - Blends with different melting rate
  - Fluff



Contiguous Solids Melting

Gauge Bands/Gels

- Contamination
  - Fibers
  - Cross contamination w/other Polymers
- Un-melt & un-mixed
  - Bad Concentrate
  - Blends with different melting rate
  - Fluff
  - Bad temperature profile

Gauge Bands/Gels

- Contamination
  - Fibers
  - Cross contamination w/other Polymers
- Un-melt & un-mixed
  - Bad Concentrate
  - Blends with different melting rate
  - Fluff
  - Bad temperature profile
  - Inconsistent granular size

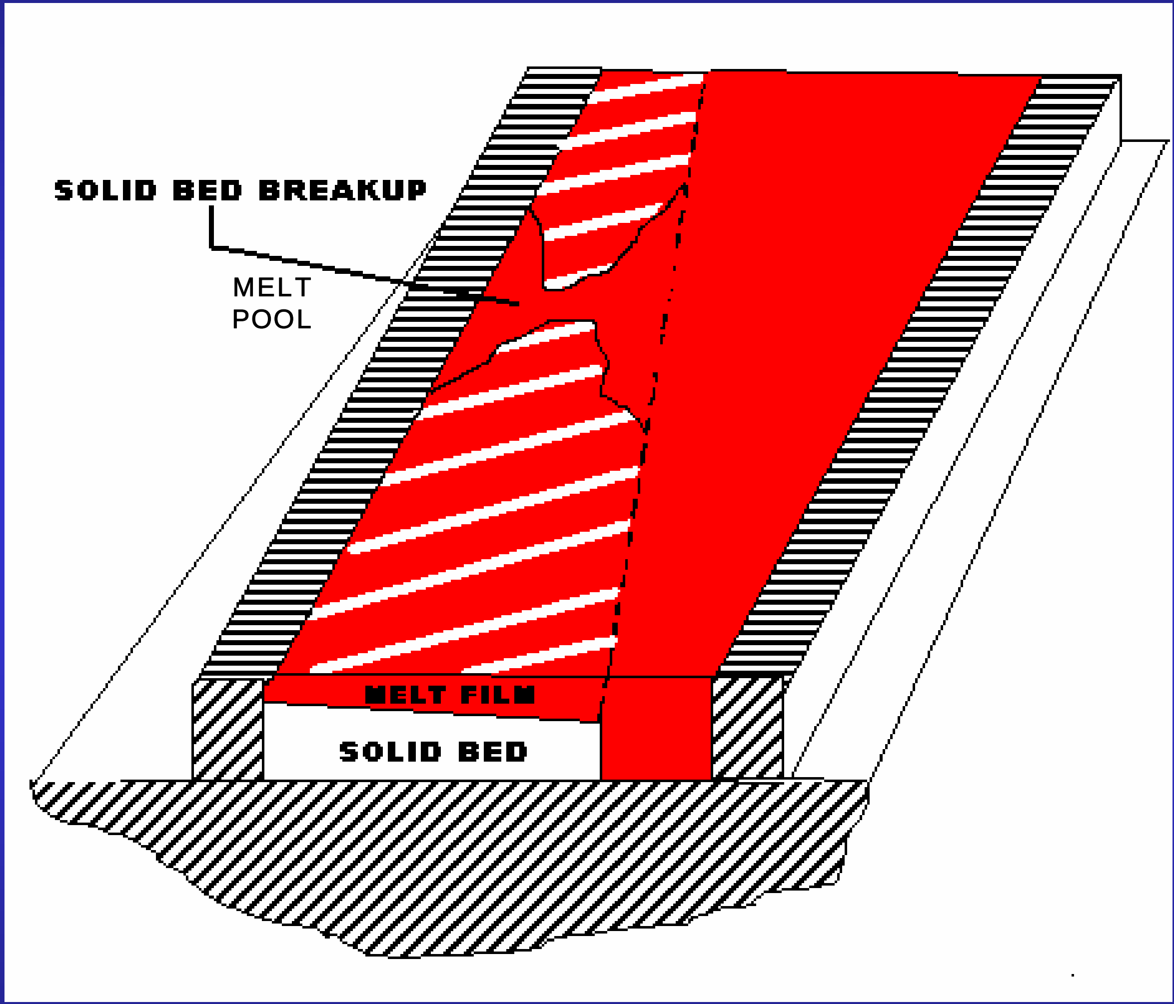
Gauge Bands/Gels

- Contamination
  - Fibers
  - Cross contamination w/other Polymers
- Un-melt & un-mixed
  - Bad Concentrate
  - Blends with different melting rate
  - Fluff
  - Bad temperature profile
  - Inconsistent granular size
  - Screw design

Screw Design

- Improper design for the resin can contribute to:
  - SBB causing encapsulation of un-melt solids
  - SBB also causes non uniform mixing history
  - Not providing enough shear to melt resin

Observed Solid Bed Break-up

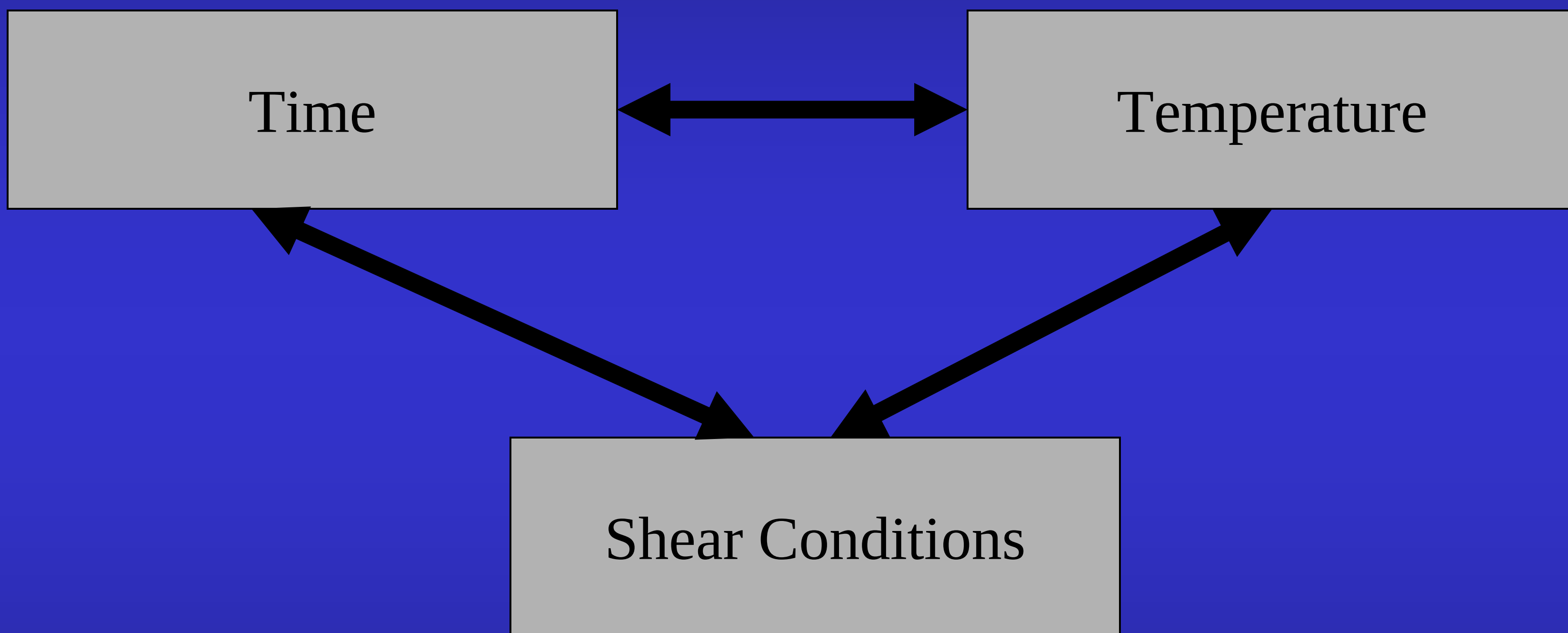


CONVENTIONAL
SCREW

Gauge Bands/Gels

- Contamination
  - Fiber
  - Resin Contamination
- Un-melt and un-mix
- Thermal degradation

Polymer Degradation is a Function of



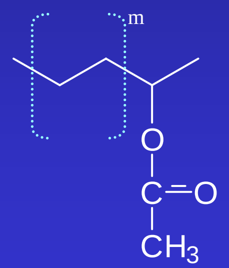
All polymers degrade as a function of time, temperature, and shear conditions

Degradation → Gels

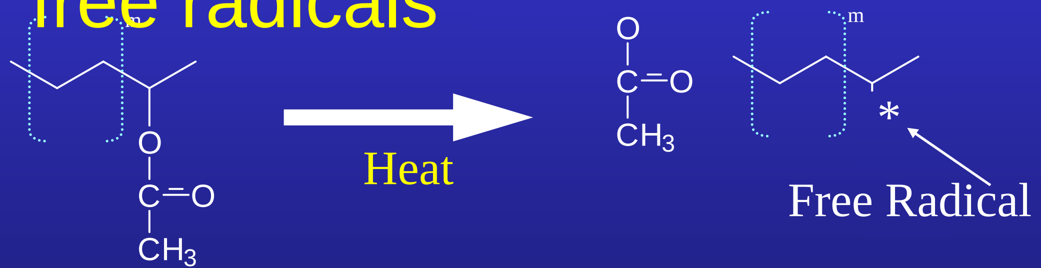
Types of Polymer Degradation

- Thermal - High temperatures
- Chain Scission - High stress fields
- Cross-linking - Un-saturation
- Oxidation - Oxygen

Example of crosslinking



- Ethylene Vinyl Acetate (EVA)
  - $-(CH_2-CH_2)_m-(CH-CH_3COO)-$
- Temperature causes decarboxylation which result in releasing Acetate, CO_2, and primary free radicals

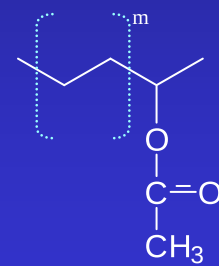


EVA Degradation

- Creates acetic acid at LDPE process temperatures.

EVA - Effect of Over Heating...

- Ethylene Vinyl Acetate (EVA)
  - $-(CH_2\text{-}CH_2)_m\text{-}(CH\text{-}CH_3COO)\text{-}$



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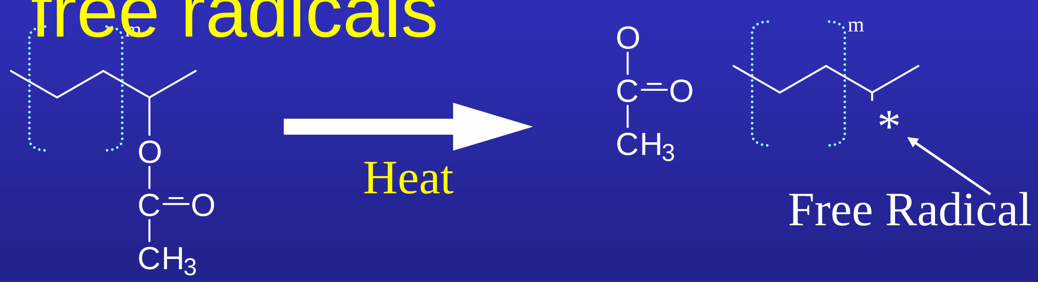
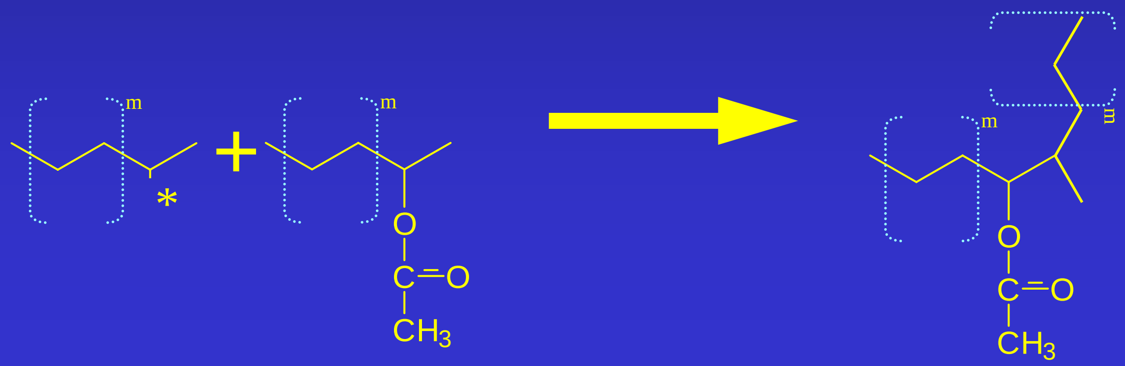
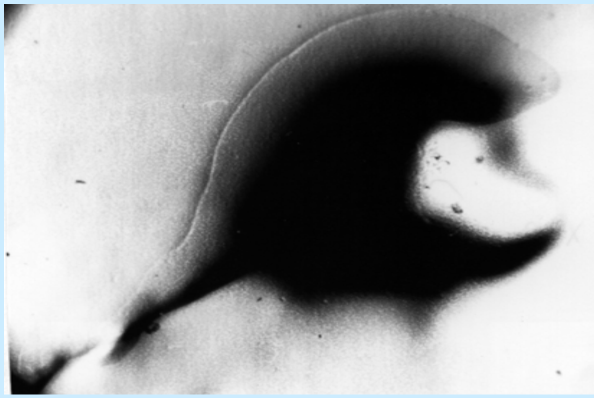
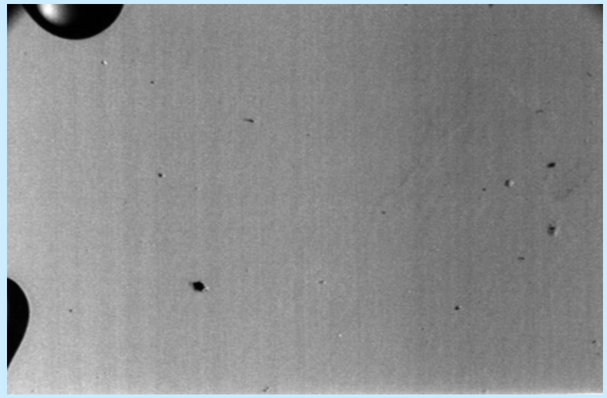


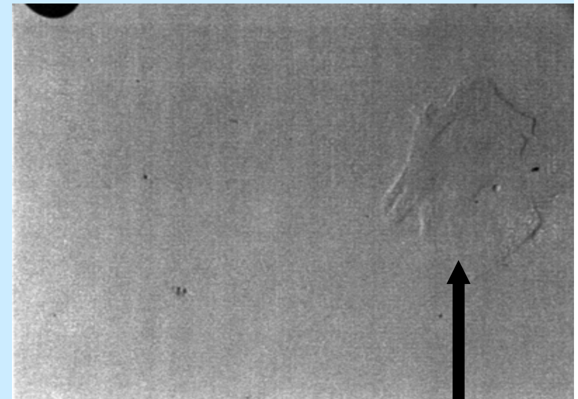
Side Reaction



Lightly Crosslinked Gel







50°C → 150°C → 50°C

Melt

Lightly crosslinked

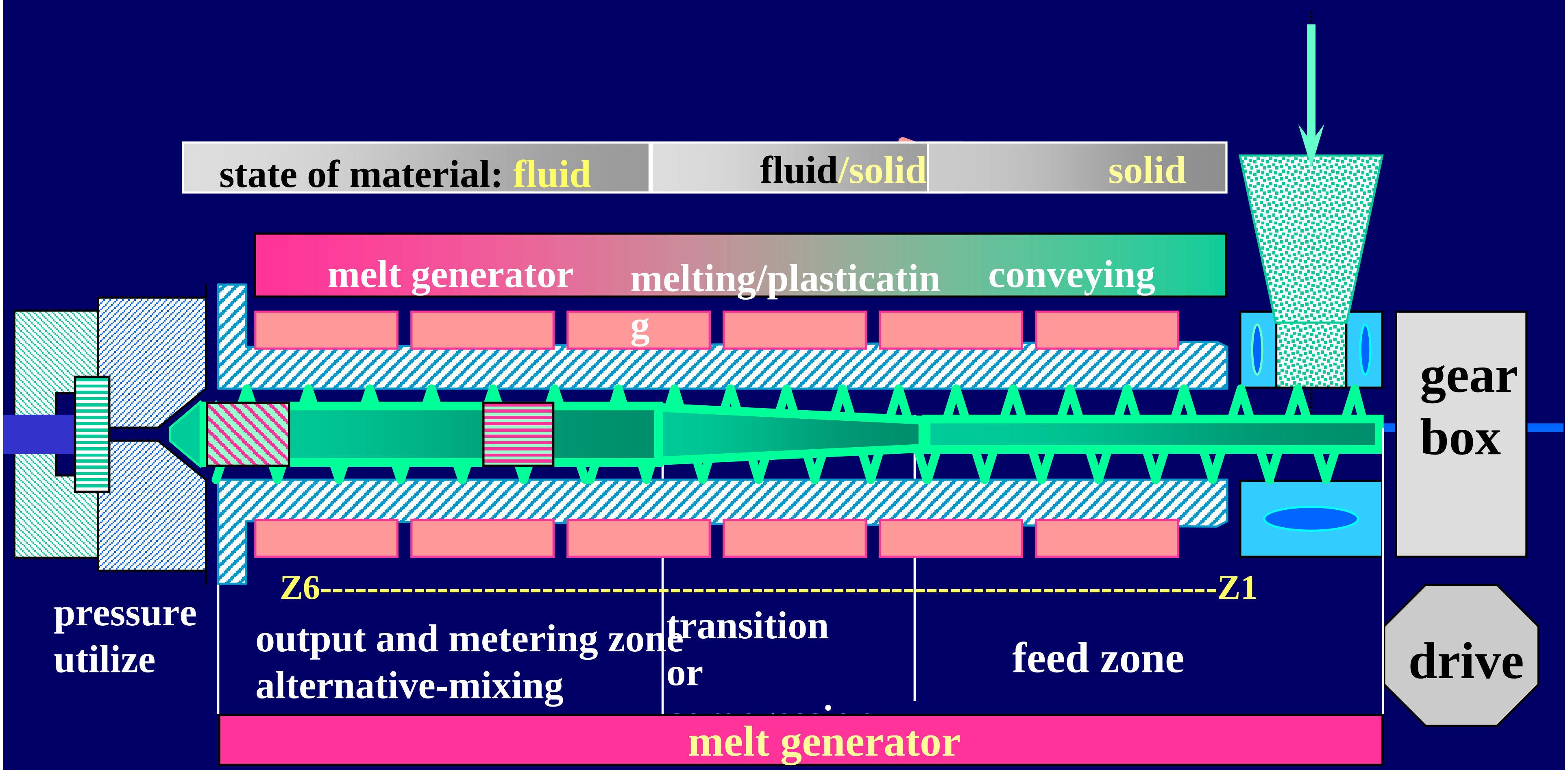
What does crosslinked mean?

- Too much energy provided to the polymer.

Too much heat

- Shear

Cross Section of Single-Screw Extruder



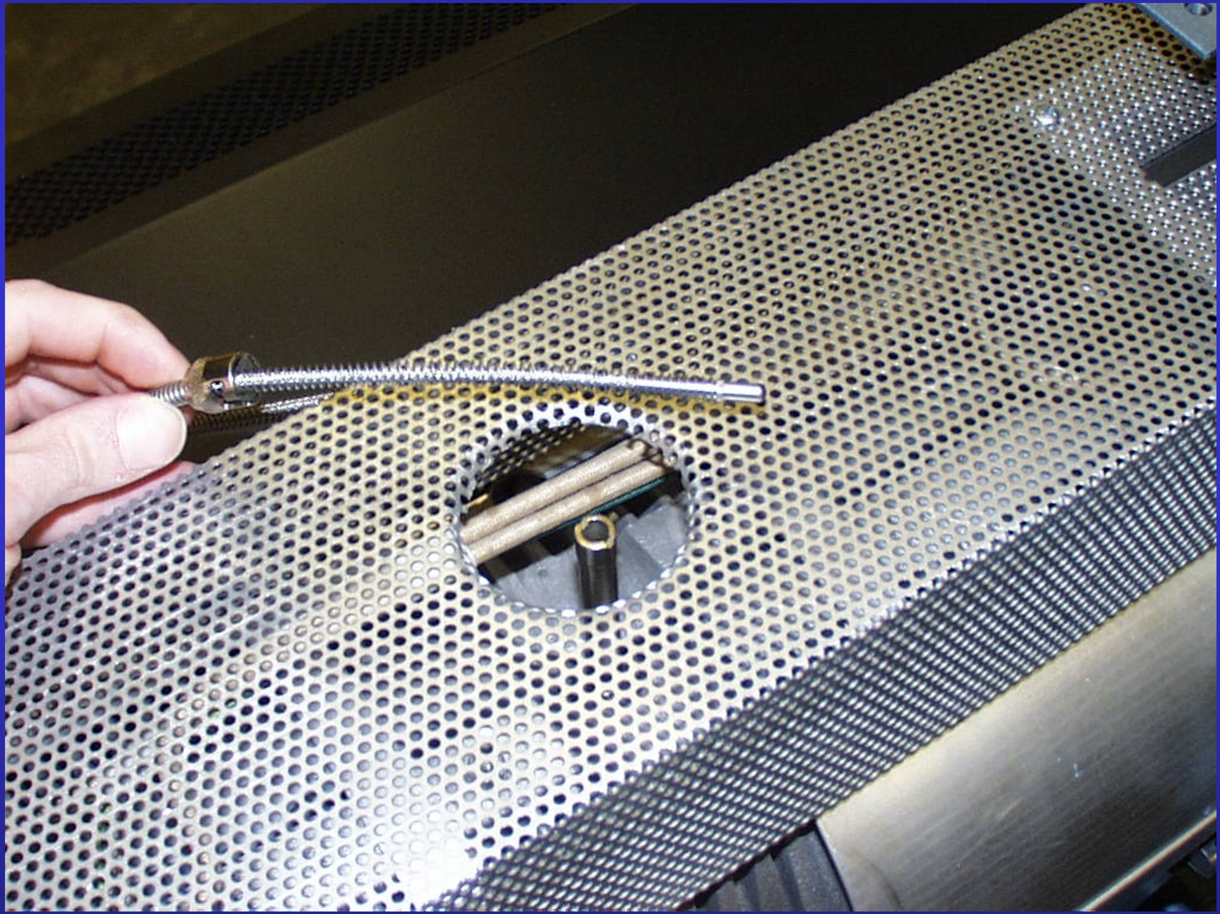
Too much heat

- Shear
- Heaters

Too much heat

- Shear
- Heaters
  - T/C not in bottom of well

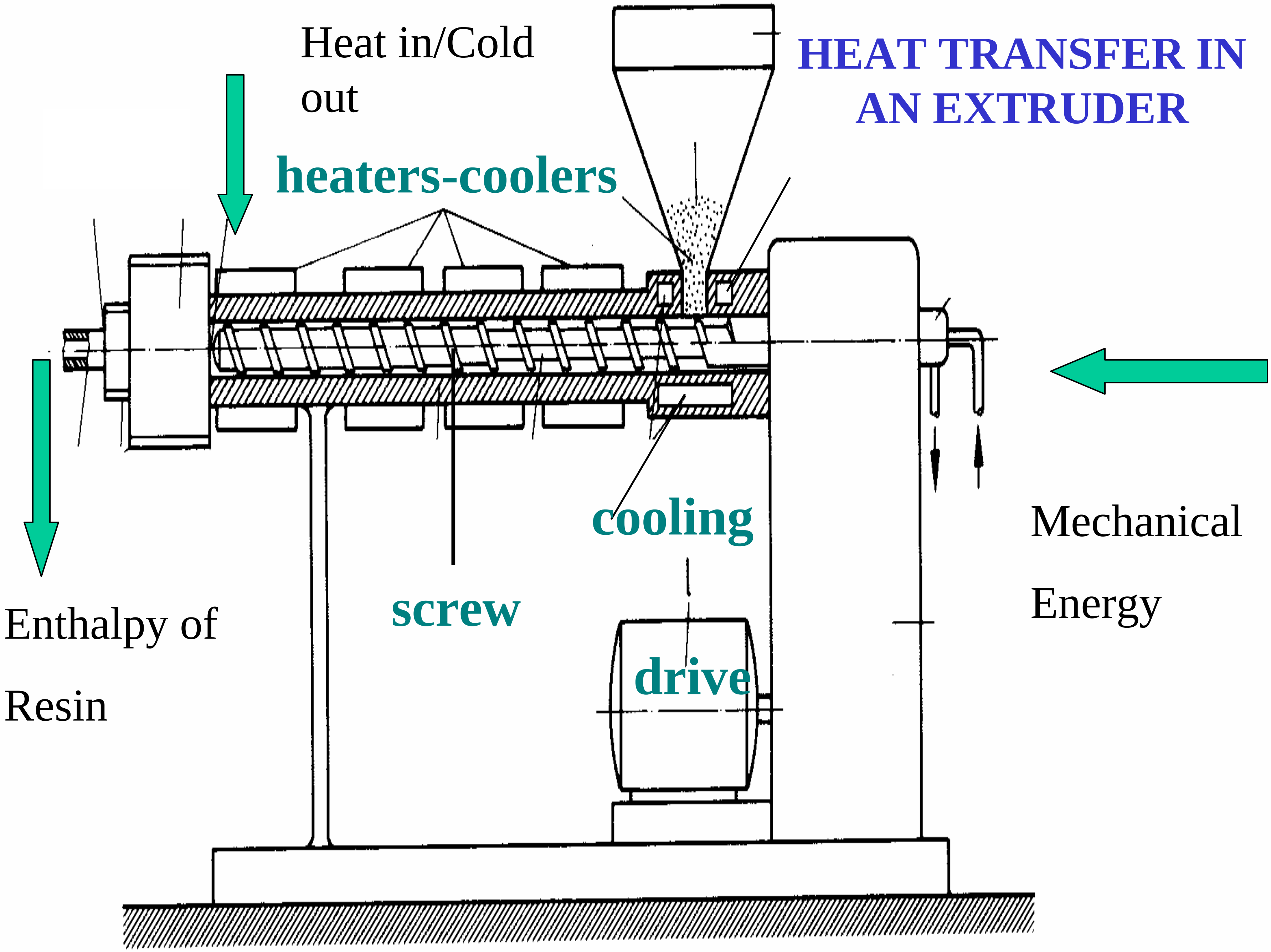
T/C Location



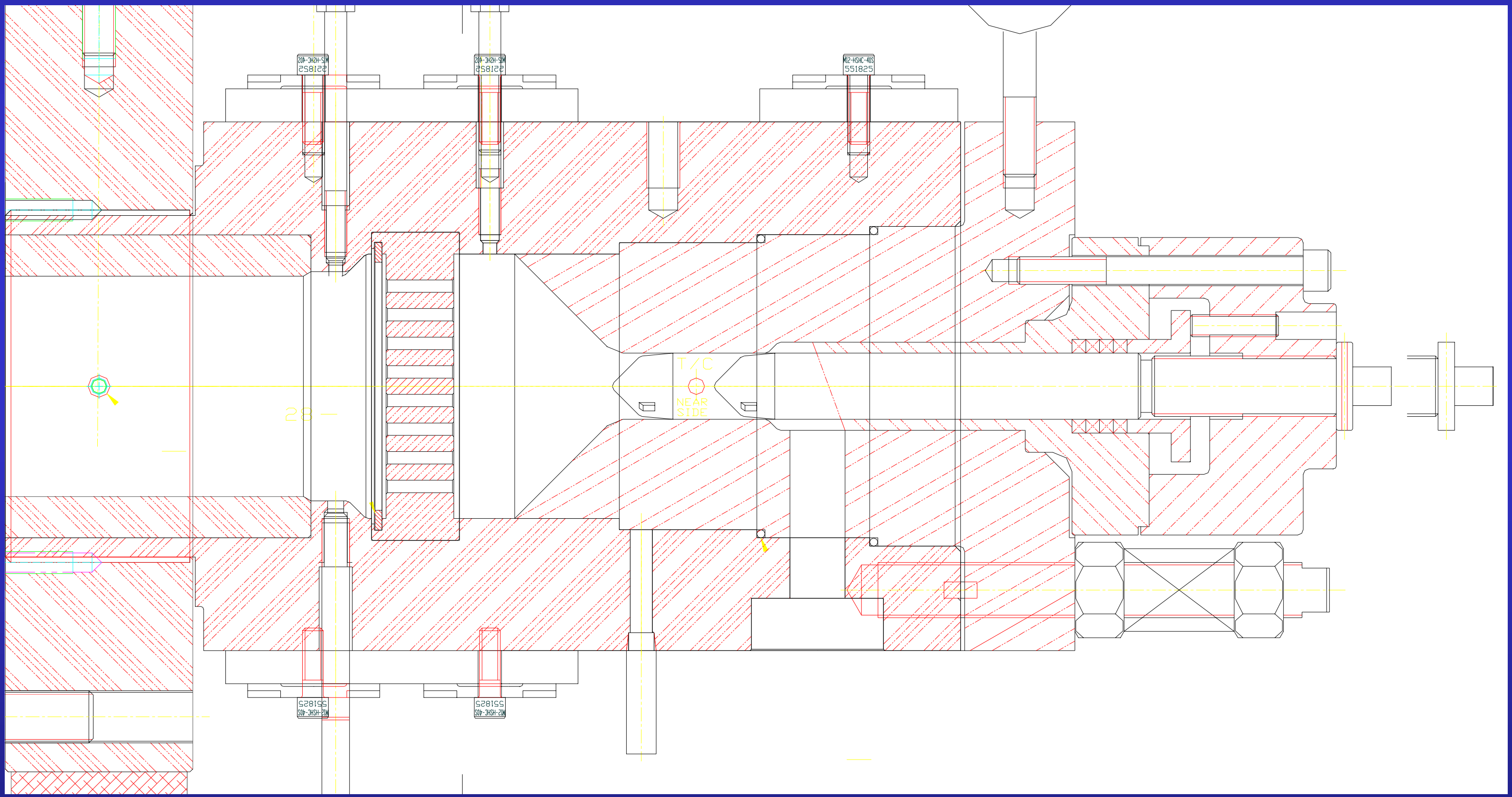
Too much heat

- Shear
- Heaters
  - T/C not in bottom of well
- Extruder Residence time

HEAT TRANSFER IN AN EXTRUDER



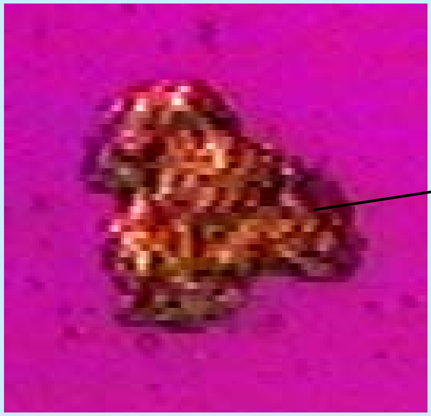
Gel Hang-up at adapter



Gauge Bands/Gels

- Fibers/Contamination
- Cross contamination w/other Polymers
- Un-melt
- Cross-Linked
- Oxidation

Oxidized Gel



Gel

Oxidized Gel at 130 C with polarized light (w/ 1/4 filter)

Oxidized Gels

- Formed very rapidly in the presence of oxygen
- Corrective Action
  - Improve stabilizer package
  - Reduce exposure to oxygen during processing
  - Reduce Temperature

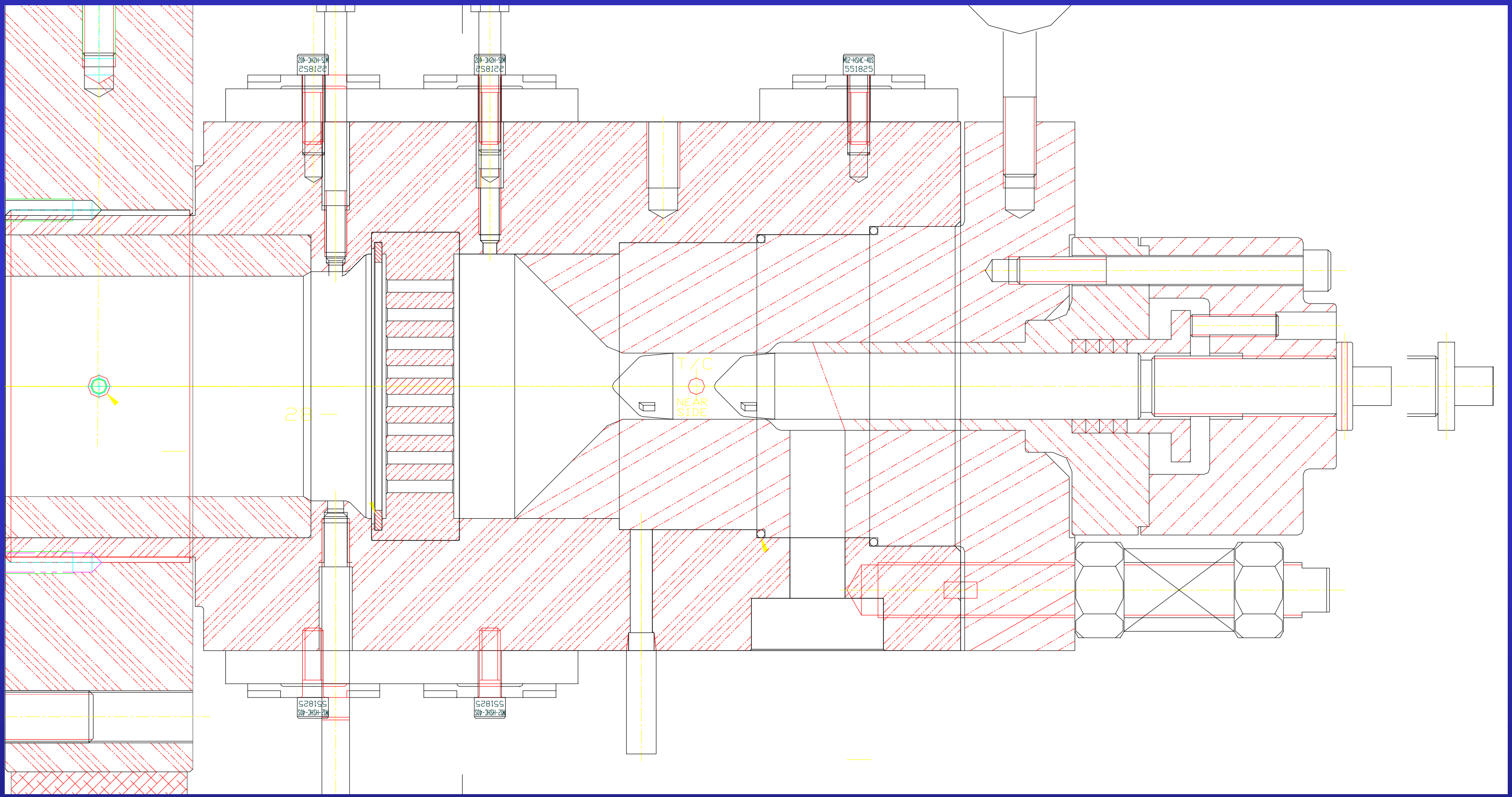
Oxidation of resin

- Incorrect shut down (not capping die)
- Incorrect start-up with cleaned die

Gauge Bands/Die Lines

- Hang up of gels
- Temperature variation
- Automatic die control
- Static Mixer position
- Hang Ups in flow path

Gel Hang-up at adapter



Solving Gel Problems

- Identify the source of the Imperfection
  – Un-melt
  – Degradation (Cross link or Oxidized)
  – Contamination (Dust, Dirt, Fiber)
  – Agglomeration (Color, Additives)
- Find the Source
  – Resin
  – Equipment
  – Blends/Additives

Tools to detect gel problems

- Hot stage microscopy
- IR analysis
- Chemical Analysis
- DSC unit

Thank You

PRESENTED BY

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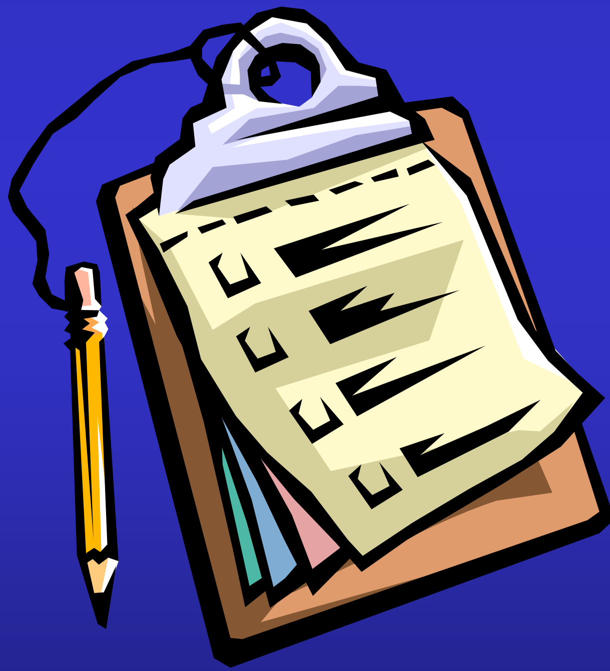
Davis-Standard, LLC



Please remember to turn in your evaluation sheet...