Plastics Failure Analysis

Dr. Ahamed Shabeer
Polymer Scientist
Bodycote Testing Group- Orange County Materials Testing Laboratory
Anaheim, California
What Does Failure Analysis Require?

- Expertise
- Willingness to Not Give Up
- Systematic Approach
- Sometimes Luck!
Than
Like
This
Failure Diagnosis is...

Causes

Techniques
Failure Analysis Protocol

- Collection of Background and Selection of Samples
- Review of Safety Considerations
- Establishment of Record Keeping
- Identification and Cleaning of Samples
- Macroscopic Examination and Analysis
- Microscopic Examination and Analysis
Failure Analysis Protocol (cont.)

- Determination of Failure Mechanism
- Mechanical Testing
- Chemical Analysis
- Stress (including Fracture Mechanics) Analysis
- Testing Under Simulated Service Conditions
- Analysis of Evidence and Formulations of Conclusions
Macroscopic and Microscopic Examination

More Experience Better Interpretation
Fractography

Accomplished using
- Naked Eye
- Magnifying Glass
- Common Light Microscopy
- Polarized Light Microscopy
- Reflected Light Microscopy
- Scanning Electron Microscope
Modes of Fracture

- Ductile Fracture
- Brittle Fracture
DUCTILE FRACTURE

Common in laboratory testing

Standard tensile test of a ductile polymer
- no stress concentration
- stretched at a constant rate until rupture
- applied stress is essentially uniaxial
- material undergoes gross plastic deformation or yielding (permanently deformed)

Fractures which occur after the applied stress has exceeded the yield stress of the polymer.
DUCTILE FRACTURE

Rare in practice

Ductile fractures rarely are subjects of failure analysis.

Maximum stress should not exceed a small fraction of the yield stress of the material.

Exceptions

Intended ductile failures such as:
tamper-evident rings found on plastic closures of beverage bottles
plastic seals on household fire extinguishers
BRITTLE FRACTURE

• Little or no plastic deformation

• No distortion or shape changes

• Material just “snapped”

• Mating fracture surfaces*

* Attempts to put the mating fracture surfaces together may introduce artifacts which can complicate the interpretation of the fracture surface
BRITTLE FRACTURE

Brittle fractures generally occur:

- at stresses well below the yield stress of the polymer.
- they are favored by high strain rate (e.g. impact)
- they are favored by low temperature, cyclic loading (fatigue), and a stress condition known as plane strain.

Since brittle fractures typically occur in the linear elastic range of the stress-strain curve, elastic stress analysis is justified in many cases.
BRITTLE FRACTURE

NORMALLY BRITTLE MATERIALS

Glass and ceramics
Polystyrene (PS)
Styrene-Acrylonitrile Copolymer (SAN)
Polymethyl methacrylate (PMMA)
Thermosetting Resins
BRITTLE FRACTURE

Even though brittle fractures are without gross plastic deformation, localized plasticity is observed on many fracture surfaces.
Failure Diagnosis

➤ Does problem really exist?
➤ What is the frequency of failure?
➤ Was the onset of the failure sudden or gradual?
➤ Did the onset occur after a design change, a change in raw material suppliers, a modification to the production cycle?
➤ Does the failure only occur with a specific product?
Determination of Failure Mechanism

- Poor Design
  - Fractography

- Non-Design Factors
  - Raw Materials
  - Compounding
  - Processing
  - Environment
Design Factors

- Related to Stresses in Application
- Can be Short-Term or Long-Term Related
Non-Design Factors

- Raw Materials
  - Resin
  - Additives
- Compounding
- Processing
- Environment
Mechanical Testing

- Short-term Properties
- Long-term Fatigue
- Long-term Creep
- Environmental Attack
Short-Term Properties

- Checked using Design Criteria
- Mechanical Property Testing
  - Tensile Strength
  - Elongation to Yield/Break
  - Impact Strength
  - Flexural Strength
  - Abrasion
- Operating Temperature
Long-term Fatigue

- Testing must relate to service
  - amplitude
  - Hertz
- Tested on material or part
- Tested in
  - Flexural
  - Tensile
  - Compression
Long-term Creep

- Requirement must relate to actual service
- Tested in
  - Flexural
  - Tensile
  - Compression
Plastic Compounds

Resin
- Type
- Grade

Additives
- Stabilizers
- Plasticizers
- Impact Modifiers
- Fillers/Reinforcing Agents
- Flame Retardants
- Lubricants
- Antiblock Agents
- Nucleating Agents
- Processing Aids
- Internal Mold Release Agents
- Colorants
- Antistatic Agents
- Blowing Agents
Compounding Variables

- Correct Composition
- Uniform Mixing
- Degradation
- Loss of Additives
- Contaminants
- Regrind
Ingredient Analysis

- Infrared Spectroscopy
- Energy Dispersive
- Gas Chromatography
- High Performance Liquid Chromatography
Environment

- Environmental Stress Cracking
- Loss of Stabilizers
- Loss of Plasticizers
- Polymer Degradation
- Thermal Aging
- Weathering
- Hydrolysis
- Chemicals
- BioProducts
Chemical Analysis

- Ingredient Analysis
- Thermal Analysis
- Molecular Weight Changes
- Microscopy
Thermal Analysis

- Differential Scanning Calorimetry
- Thermogravimetry
- Thermal Mechanical Analysis
- Dynamic Mechanical Analysis
Processing Causes of Degradation

- Inadequate Predrying
- Processing Conditions
  - Temperature
  - Time
  - Shear
Molecular Weight Changes

- Dilute Solution Viscometry
- Gel Permeation Chromatography
- Melt Rheology
- Sol/Gel Content
Other Worries

- Processing Defects
  - Weld Lines
  - Shrinkage
  - Voids

- Crystallinity
  - Degree of Crystallinity
  - Morphology
  - Distribution

- Residual Stress in Molded Parts
- Degree of Polymer Orientation
- Degree of Crosslinking
- Degree of Cure
Stress Analysis

- Can be analyzed using FEA
- Many times needs a different perspective

Finite element model of a transit bus

Plastic bottle weight reduction
Case 1 - Infrared Spectroscopy
Epoxy Adhesion Failure Problem

- A 2-part epoxy had been successfully used to bond transistors in hearing aids built into eye glasses.
- New problem with epoxy bond failing.
- Had the correct mix ratio been used?
- Specification: 70/30 resin/hardener.
A small flake of the adhesive was removed under a microscope.

Micro-FTIR spectrum recorded.

Resin/hardener mixtures of 60/40, 70/30 and 81/19 were made and cured.

Recorded FTIR spectra of neat resin, neat hardener and the three mix standards.
Spectrum of Resin and Hardener

Terminal Epoxy Group at 917 cm\(^{-1}\)

RESIN

HARDENER
Spectrum of Different Ratios

60/40 MIXTURE  70/30 MIXTURE  81/19 MIXTURE
Spectrum of Epoxy from Transistor

EPOXY REMOVED FROM TRANSISTOR

917 cm$^{-1}$
Could distinguish mix ratios using relative intensity of resin peaks at 2965 and 2873 cm$^{-1}$ and hardener peaks at 2922 and 2952 cm$^{-1}$.

Sample from transistor displays correct 70/30 mix ratio and no unreacted epoxy at 917 cm$^{-1}$.

Adhesion failure – likely surface preparation problem.
Case 2 - FTIR
PP Regrind Problem

- Molder uses outside source for polypropylene regrind.
- All colors and mixtures of colors are acceptable.
- Molder was concerned about possible contamination of such mixtures.
- Molder submitted a bag of regrind for analysis.
Sample consisted of small pieces of 13 different colors and shapes.

A typical specimen of each type was isolated and identified by FTIR.

Four (4) of the 13 types were not a PP.

Contaminants: PE, plasticized PVC, Nylon and Wood Chips.
Case 3 - Infrared Spectroscopy
Cracking HIPS Door Liners Problem

- Refrigerator manufacturer had a warehouse full of refrigerators with cracked HIPS door liners.
- Cracked liners were submitted for failure analysis.
Cracking appearance was consistent with Environmental Stress Cracking.

Panels had residual stresses from thermoforming.

Cracking located where panel touched rubber sealing gasket expected to contain extending oils.

Cracks contained visible oil.
Under a microscope, a needle was used to transfer oil from a crack to a salt plate.

Extending oils in a gasket were isolated by MEK extraction.

FTIR spectra of both oils were recorded.

The spectra did not match.

Oil from the crack had more peaks.
HIPS Door Liners
Identifying Additional Oil

- Spectral subtraction of the gasket oil from the crack oil yields a spectrum for an unsaturated ester such as a vegetable oil.
- Further checking with plant personnel revealed that they had started to wipe the gasket with corn oil to eliminate sticking.
- Cracking caused by corn oil application.
Case 4 - Field Returned Instrument Panels PC/ABS
Failures observed in instrument panels within two years of installation.

Exorbitant cost of replacements as failures occurred within the warranty period.
Crack Propagation Mechanism
Microscopic Examination

- Crack initiation from the exterior corner edge near ventilation grill
- Smooth crack origin indicates low level of stresses
- Incremental crack propagation due to cyclic stresses as suggested from striations
- Fast crack growth when critical size is reached
**Izod Impact Strength**

- Conditions
  - Tested in controlled environment laboratory
  - 2 inch-pound hammer
  - Tested according to ASTM D256

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<table>
<thead>
<tr>
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<tbody>
<tr>
<td>Fractured Panel</td>
<td>0.42 ft-lbs/inch</td>
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<tr>
<td>New Panel</td>
<td>1.24 ft-lbs/inch</td>
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Melt Flow Index

MFI ASTM D1238
Conditions: 250 C
1.2 Kg load

Failed Panel 14.2 g/10 minute
New Panel 4.3 g/10 minute

Each data point is an average of 3 experiments
FTIR Analysis

Bodycote Broutman
Dashboard Vent
Wed Jun 19 10:51:43 2002

Sample 2 Liquid Residue From

Wavenumbers (cm⁻¹)

% Transmittance

4000 3500 3000 2500 2000 1500 1000 798

1260.076 1090.544 1018.427 986.462
FTIR Analysis

Silicone oil - ESC agent
Case 5 - Streaks in Plexiglas

- **Problem:** Microwave door panel of Plexiglas (PMMA) has white streaks
- **Possible Cause:** Contamination
- **Technique Used:** Microscope FTIR
Plexiglas Sheet

Contaminated Streaks
Case 6- Which PP is this?

Problem: Manufacturing Plant has transferred material to unlabeled Gaylord. They only have two types of PP but the materials are different.

Technique Used: Thermal Analysis - DSC
# Distinguishing Two Similar Polypropylenes

<table>
<thead>
<tr>
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<th>General Purpose</th>
<th>Improved Processability</th>
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<tbody>
<tr>
<td>Melt Flow</td>
<td>12</td>
<td>12</td>
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<tr>
<td>Density</td>
<td>0.903</td>
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<tr>
<td>Izod</td>
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<td>Elongation</td>
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<tr>
<td>Rockwell R</td>
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<td>103</td>
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</table>
DSC on Polypropylenes
Case 7 - ABS Knob Crack - ESC
Case 7 - ABS Knob Crack - ESC

Inside surface - oil near the crack
Case 7 - ABS Knob Crack - ESC

FTIR of liquid residue in cracks - silicone oil
Case 8 - PE Pipe Failure

- Ductile failure of polyethylene gas pipe in service in Arizona
- Petrochemical odor
- Litigants claimed byproducts in natural gas
Case 8 - PE Pipe Failure

SPME sampling and GC-MS analysis detected large number of aromatic and naphthenic compounds, including PAH’s, consistent with diesel fuel exposure.

Fuel plasticized PE, leading to ductile failure.
Bodycote Advantages

- Multidisciplinary Staff
- Staff Expertise
- Industry Experience
- Problem Solving Experience
- Extensive and Diverse Laboratories
- ISO 17025
- Laboratories Accredited by A2LA & ICBO, NADCAP for non metallics
- Customized Reports with Complete Documentation
- Approved by Aerospace primes
Major Areas of Expertise

Failure Analysis
Expert Witness
Polymer Analysis
Materials Testing
Product Design
Microscopy
Piping Products
Polymer Characterization
Composite Characterization
Process Troubleshooting
Product Testing/Development
Stress Analysis/Design
Thank you for your attention........

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