Failure Analysis

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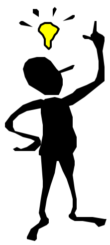
I have been accomplishing failure analysis on polymeric products for over twenty years. Usually the conversation with the engineer who is in charge of the investigation begins the same.

> *"We are recently having failure on a product that has been working for years. We don't understand the failures because nothing has changed."*

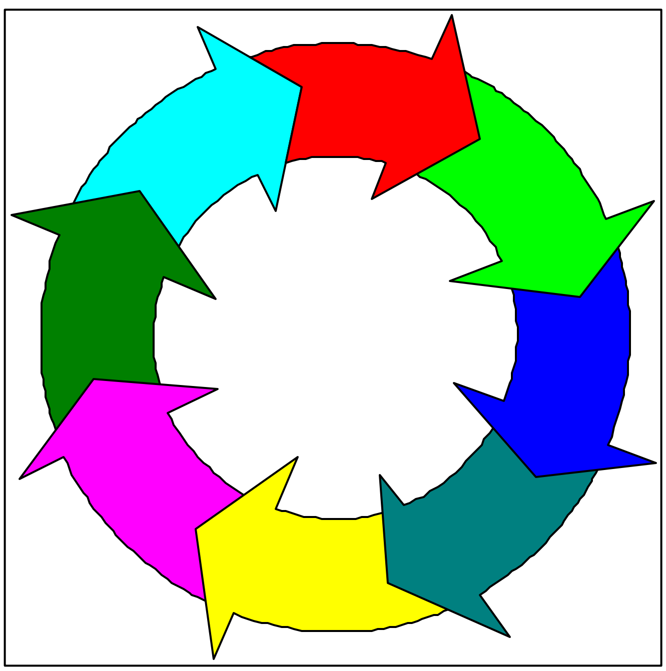
Failure Analysis requires:



- ✓ Expertise
- ✓ Perseverance
- ✓ Systematic Approach
- ✓ Luck

Since I haven't found the formula to guarantee luck (if I had I would be in Vegas and not working in the Plastics industry) I will focus this discussion on the Systematic Approach.

Failure Analysis is more like this:

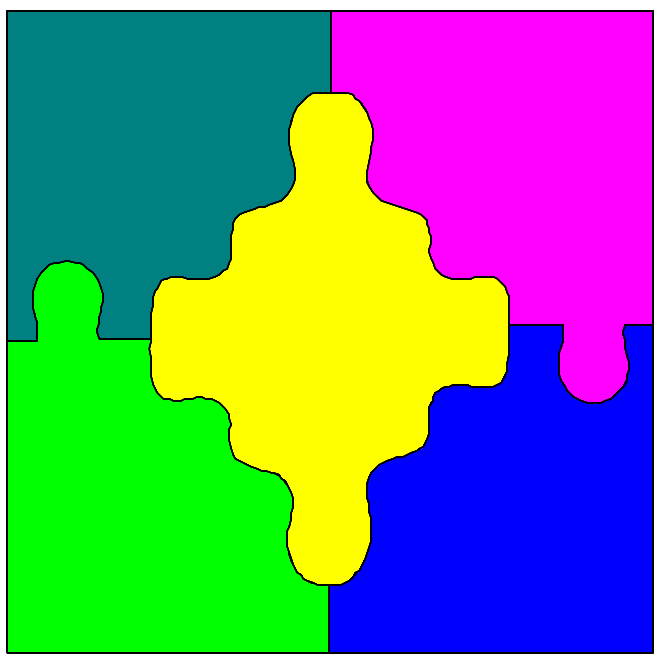


Failure Analysis Protocol

1. Collection of Background and Selection of Samples
2. Review of Safety Considerations
3. Establishment of record keeping
4. Identification and Cleaning of Samples
5. Macroscopic Examination and Analysis
6. Microscopic Examination and Analysis
7. Determination of Failure Mechanism
8. Mechanical Testing
9. Chemical Analysis
10. Stress (including Fracture Mechanics) Analysis
11. Testing Under Simulated Service Conditions
12. Analysis of Evidence and Formulation of Conclusions

Failure analysis involves a systematic approach to identify possible causes and utilize analytical techniques to pinpoint the exact cause(s). You must not only know how to diagnosis the failure mode, but also to determine the best way to accomplish this diagnosis. Listed above is the twelve-step process used for failure analysis. (Twelve step process – it's sort of a catchy phrase.)

Than like this:



Step 1 Collection of Background Data and Selection of Samples

Some of the questions that need to be asked in Step 1 are:

Does the 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, or a modification to the production cycle?
Does the failure only occur with a specific product?

Failures can be categorized into two categories:

Design Factors

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

It is amazing to think that all of the product failures can be classed into these categories. However from personal experience, failures are often a combination of factors, which is why I showed the above boxes overlapping. A good example is the recent Firestone tire failure. Although the discovery is still underway, several factors are being considered including design (of tire and vehicle), processing (only happens at one plant) and compounding (use of inferior material).

The engineer closest to the failure usually accomplishes steps 1 – 4 on the Failure Analysis Protocol. Frequently Failure analysis reaches a stumbling block concerning analysis when it comes to sample collection. Many companies keep retains of all production, but in these times of cutting costs, frequently this process is short-changed. Either the exact manufacturing date or location are hard to pinpoint, or retains of unused stock are unavailable, or even the exact material used for production is hard to determine. This stumbling block often leads to incomplete failure identification.

It is very important that record keeping be accomplished in an organized manner. The thought that this might be involved in a legal proceeding should be kept in the back of everyone's mind. Remember the Law and Order TV court cases that have been won/loss because of poor retention of samples or mislabeling.

Step 2 - Review of Safety Considerations

Frequently failures involve materials that have been exposed to hazardous chemicals. Care should be taken to identify those chemicals and protect that analyst from exposure. This might involve everything from rubber gloves to full-protection suits. I would recommend that anyone involve with failures wear gloves and particle masks while working with the samples.

Step 3 - Establishment of record keeping

It is also important that record keeping be accomplished in an organized manner. The thought that this might be involved in a legal proceeding should be kept in the back of individuals mind. A notebook or database with all samples collected and pertinent information should be kept. Information noted should include but not be limited to:

- ✓ Where was sample collected.
- ✓ Date and time of collection.
- ✓ Number of pieces in sample if broken.
- ✓ Any manufacturing marks on sample.
- ✓ Safety considerations.
- ✓ Any observations about the environment.

Lee Nylander, of Bodycote Broutman, was recently involved in a failure analysis case that would have been unsolvable without this all important record keeping. The failures were intimately tied to the number of hours that the units were in service. Therefore this information was important to capture the failure mode and also predict the service life.

Step 4 - Identification and Cleaning of Samples

It is important that all samples be identified. This is easily accomplished if the record keeping has already been started. However there are several things to keep in mind during the identification:

1. Use non-water soluble ink on all tags.
2. The best markers to use on non-black parts are "Sharpie" type. Use a fine point if available.
3. If the sample is black a good marker choice is either one with gold or silver ink.
4. Another choice for black samples is white-out
5. Mark all pieces of samples.
6. If fluids are present seal the samples in glass jars. (plastic bags will not necessarily keep all fluids intact.)

Be careful when cleaning samples. When in doubt don't clean. A famous example of excessive cleaning can be found in tank liners used to transport liquids either via rail cars or tractor-trailers. It is common practice for these to be steam cleaned before examination of the failures. This is claimed to be a safety consideration, since these are commonly used to carry acids. However the steam cleaning removes all traces of the liquids, both the anticipated and the possible culprit of the failure.

Step 5 – Macroscopic Examination and Analysis

It is important that the samples and as much of the system as possible is examined during a failure analysis. I strongly recommend a visit by the investigator. Frequently several hypotheses will come out of these visits that had not been discussed before. Or sometimes a visit will change the area of initial investigation. One of the lengthy cases that I worked on was one where I was just sent very small pieces of samples and told to identify the plastic. The assumption had been made that the failure was because the wrong plastic had been used. I eventually confirmed that the right plastic had been used, but since that didn't solve the failure analysis many iterations of samples and analysis had to be accomplished before a conclusion was made. A simple trip to the failure would have saved many months and some monetary investment.

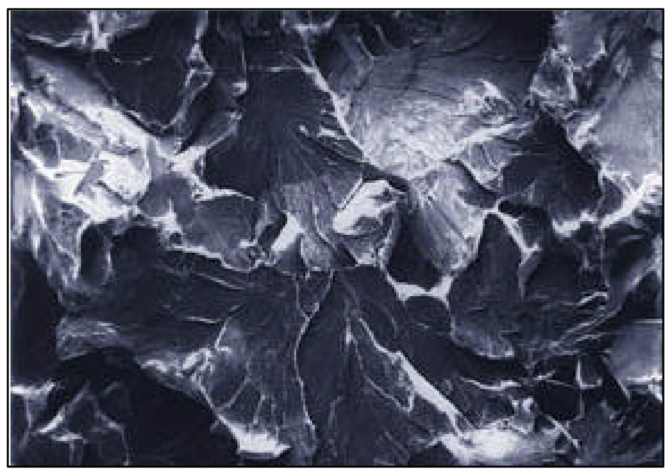
Step 6 - Microscopic Examination and Analysis

Fractography is the visual and microscopic study of fractured surfaces and is commonly used to look at failures. The more experience you have with fractography, the better the analysis.

There are two classes of Failure, Ductile Failure and Brittle Failure. Ductile failure is the type of failure that happens over time. Visually it looks more like a topographical map of well-worn old mountains. Brittle failure is a sudden failure and the surfaces are sharp and angled.

Fractography can be accomplished using:

The naked eye
Magnifying Glass
Common Light Microscopy
Polarized Light Microscopy
Reflected Light Microscopy
Scanning Electron Microscopy



An example of a Brittle Failure

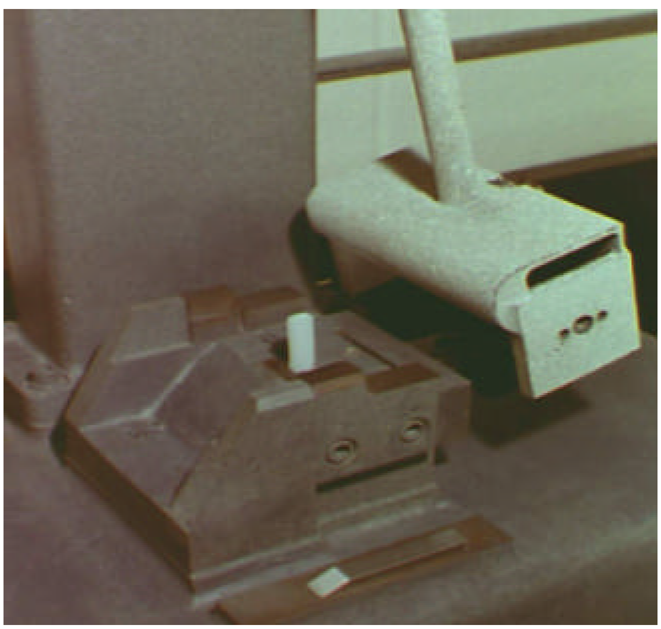
Step 7 - Determination of Failure Mechanism

It is important that the scientific method be used when developing theories for failure mechanisms. This is one of the best times to utilize teams to brainstorm possible mechanisms. The members of the team can list all the possible causes of failures and then rank them in order to investigate. You shouldn't base your ranking on costs to perform the analysis to see whether that is the cause of failure, but the costs to fix the problem. For example, there might be an inadequate amount of stabilizer in a plastic system. It might costs $3000 to investigate this, but only $1000.00 to fix a potential liability of $100,000.00. The investigation would be well worth the investment.

After you have your listing of failure mechanisms, a systematic approach should be undertaken to eliminate the ones not leading to the failure. If you have done your homework and are lucky you will narrow down the mechanisms list immediately.

Step 8 - Mechanical Testing

Determination of short-term mechanical properties is very critical if your failure is a brittle type failure. You must enlist the help of engineers to determine what are the stresses, in stretching, bending and compression that the part is seeing during service. You can then determine if the part meets these requirements.



A sample being impacted

In almost all cases samples can be machined from the actual part and tested. This should be done wherever possible. A second choice would be to have material from the same lot molded into specimens, and a third choice would be check the properties using a general lot of material. Not preferred, is relying on supply information concerning the mechanical properties of the material.

A part being machined for strength testing

Step 9 – Chemical Analysis

Usually, more money is spent in chemical analysis of failures then in any of the other steps. Plastic materials are almost never just one “material”. Probably the purest material that is used today is Teflon.

Plastics consist of a “type of resin”, which then can be classified by different grades. A type of resin is “polypropylene.” However polypropylene is sold in many different grades. These different grades of materials can have very many different properties. In addition, almost all plastics have additives that are non-plastic (polymeric). These additives are put in to:

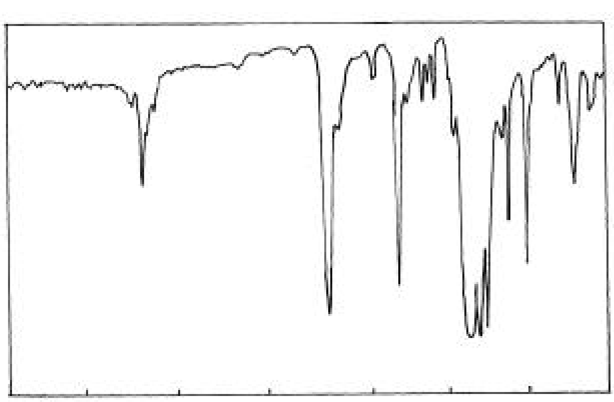
Stabilize
Improve Processing
Improve impact characteristics
Make the product cheaper
Reinforce the properties
Increase Flame Retardance
Decrease the surface friction
Color
And Many Others

All of these additives can contribute to failure, or prevent failures. I have worked on several cases where what everyone assumed to be a benign additive, affected the life of the product accomplished to determine what is happening chemically in the area of failure.

There are many analytical techniques available for the chemist to use. Most of the instrumentation is expensive, but analysis can be accomplished very quickly, normally in a day or two.

An FTIR with Microscope Attachment

A common technique is Fourier Transform Infrared. This technique provides a "fingerprint" of the material in question, which then can be matched to controls to determine the chemical composition of the material. Organic materials are more easily defined then inorganic, but the technique is used universally. Of particular importance to failure analysis is the ability to perform FTIR on a microscopic sample.



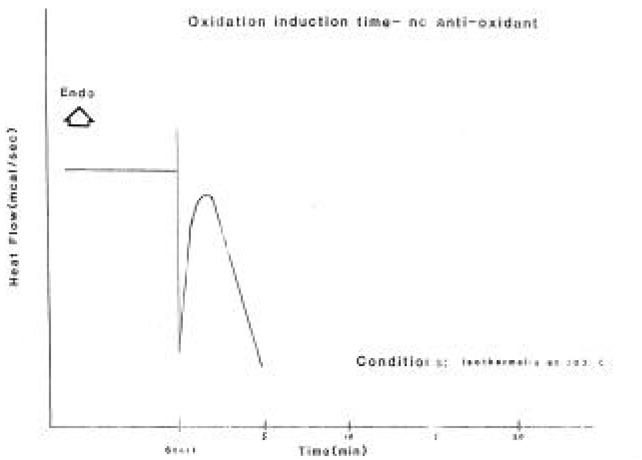
An Example of and FTIR Fingerprint (Spectra)

Another set of useful techniques for failure analysis is thermal analysis. Many failures are related to the material being used at either too high (or too low) a temperature. Probably the most notable and costly type of failure related to service temperature was the Challenger disaster back in 1986.

Differential Scanning Calorimetry can be used to determine the location of transition temperatures for polymers. These transition might be melting, degradation or else more specific transition to the material. The sample is tested in a static mode and the information is charted in a graph form. Dynamic Mechanical Analysis can also be used to determine this data, but the sample is then tested in a dynamic mode. The transitions do vary based upon whether the samples are tested dynamically or statically. From a failure analysis point of view, if the sample is being used as a vibration isolator on an automobile, then it should be tested in a dynamic mode. However if you were investigating the upper operating temperature of food storage containers, then a static mode would be preferred.

An analyst measures a sample for DMA

Another thermal analysis technique utilizes Differential Scanning Calorimetry to determine the stability of the plastic to oxygen. Oxidation is a well-known method of plastic failure. It can take many forms but is most widely associated with Polypropylene. Have you ever had a plastic part on your car look bleached? That is oxidation.



An example of an Oxidative Induction Curve

Many other Chemical techniques are used. Usually the focus is placed on “Did I get what I ordered.” That frequently means accomplishing a deformulation on a material. Most materials can be deformulated to provide a good guesstimate of the ingredients. There are a few tricky materials, which can confuse the uninitiated. Silicon rubber fooled me the first time I encountered it. The problem is that the polymeric (organic) component also contains Silica, which is frequently used also as an additive. To separate the polymeric from the additive can be very difficult.

However, very few times have I been stumped completely, and with much thought and input, a compounding problem can be found using chemical analysis.

Step 10 - Stress (including Fracture Mechanics) Analysis

Design factors contribute extensively to failures, but non-design factors very often are more important. The incorrect material is sometimes chosen for an application. It might not meet the short-term strength requirements, the long-term fatigue or creep requirements or been in an environment that is too harsh for the material.

Stress analysis uses computations to determine whether the material has enough strength to meet the design requirements. This used to be calculated using engineering principles. It still is calculated using these principles, however high powered computer crunch the numbers. In order to perform the calculation, a part design must be inputted to the software. The software will then divide the part into finite elements (small blocks) and determine the stresses on those elements (individual blocks). These stresses are then combined to determine whether the part can meet the requirements.

For example, I was sent about twenty cup holders from one automotive supplier. The car manufacturer had required a certain price for the cup holder, which dictated that use of a relatively weak material. The cup holders had broken during normal use, because the material could not handle the banging around that it received. We accomplished a microscopic examination on the parts to determine the mode of failure. Although stress analysis had been accomplished during the design phase of the part, the value of the stress (banging around) had been underestimated. By increasing the stress values in the analysis they were able to determine the areas of possible failures. These areas matched exactly what we had observed in the failed returned parts.

Step 11 - Testing Under Simulated Service Conditions

It is commonly this step that is left out of the failure analysis process. Many investigators formulate the hypothesis, but don’t check whether that hypothesis is correct by simulating the proposed method of failure. If you do not confirm your hypothesis, then frequently it will come back to haunt you with additional failures.

One failure analysis that I worked on early in my career involved contamination of steel sheet during the forming process by the rubber from rollers. We thought that we had isolated the problem to only one set of rollers, which were then replaced. The failure came back and we discovered that it wasn’t a bad set of rollers, but instead an environmental effect from various solutions they were exposed to. A simple life prediction study of the rubber in the chemical environment would have pinpointed the problem thereby not causing the wasted replacement of rollers that were bound to fail again.

Testing of material under stresses in a controlled environment

Step 12 - Analysis of Evidence and Formulation of Conclusion

Every part of the analysis should be documented including the final conclusions. The tendency is to rejoice that the problem was solved, but to not write down the solution. Unfortunately it seems as if sometimes we are destined to make the same mistake over and over again. With the amount of early retirements/golden handshakes increasing this documentation becomes very important.

Make sure that there is a final meeting with all parties involved so that everyone understands the process that was used and the conclusions. Wouldn't we all hate to see another Challenger accident because the file on the failure analysis was incomplete and the same nonfunctioning material was used because the next project went to the lowest bidder? [For photographs of the challenger disaster go to www.rjsullivan.com/chllngr/#.]